

# Chemical & Process Engineering

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## Topics of the Month

### Productivity in heavy chemicals

ON March 6 a team representing the heavy inorganic chemicals industry of the United Kingdom sailed for a six-week tour of the U.S. to study the productivity of the equivalent American industry. This is the third chemical productivity team to go to the U.S., the other two being concerned with fertilisers and pharmaceuticals respectively.

One of the first concerns of the team will be to define and measure productivity. The means of measurement adopted by the team will arouse considerable interest because, of course, productivity, in the sense of output per man-hour, is a most difficult thing to measure in an industry which produces huge volumes of product in enormous plants quite often supervised by no more than two or three men. The compilers of the Association of British Chemical Manufacturers' 'Report on the Chemical Industry,' published in 1949, took the term productivity to mean the added value per man-year of an industry's products, *i.e.* the total value less the value of the raw materials used. On this assumption it was stated that the chief means of increasing productivity in the chemical industry was by improving processes and products. Few will quarrel with this view; the fact that a change from a batch to a continuous process can often treble productivity is sufficient proof of its validity. Undoubtedly, therefore, the team will wish to study how

improvements in processes have increased productivity in the U.S. Indeed, the emphasis on this aspect of productivity is evident from their terms of reference which, besides the definition and measurement of productivity, include:

- Factory and plant layout;
- Constructional materials for plant;
- Instrumentation and automatic control;
- Mechanical handling of materials;
- Treatment and disposal of effluents;
- Standardisation of equipment and products;
- Organisation of plant maintenance;
- Supervision and cost control;
- Labour recruitment, training, promotion, shift-working, incentives and the like;
- Safety and welfare.

Most of the foregoing aspects of productivity are the direct responsibility of the chemical engineer, particularly in the case of heavy chemicals, in that the fundamental reactions involved in making most of them have been known for decades and such improvements as have been made have been in the re-design of plants in accord with advances in chemical engineering knowledge and practice. The Americans have a longer tradition of a distinctly chemical engineering approach to chemical manufacture than any other nation and it will be instructive to see through the

expert eyes of the team how their processes and techniques compare with those in Britain.

It is to be expected that in factory and plant layout, mechanical handling, standardisation of equipment and products, plant maintenance and supervision, and cost control the team will learn much from the Americans. In other fields, particularly safety and welfare and what may be called the human side of the industry, the Americans may well learn something to their advantage from the British.

As measured by the acid test of the prices of its products the British chemical industry, particularly heavy inorganics, has a good record. Between 1938 and 1948, for example, the price of light soda ash rose only 26% and that of sulphuric acid 69%, compared with a general rise in wholesale prices of 124%. The heavy chemical industry is absolutely basic to the national economy and it is imperative that its productivity should increase and its prices remain low. Everything that can be learned from the Americans to foster this objective will be welcomed by all progressive chemists and chemical engineers.

### **Safe repairs of oil drums and tanks**

THE repair by welding, etc., of drums and tanks which have held inflammable liquids is governed by strict safety precautions; the combustible materials must be removed by steaming or boiling, or CO<sub>2</sub> must be added to make the atmosphere inside the vessel inert. Usually these precautions are rigidly observed in the case of liquids such as petrol which have low flash points. With oils of high flash point such as kerosene, cod-liver oil or linseed, it has been assumed that such precautions are unnecessary. This belief has led to disaster, for explosions have occurred when a flame has been applied to tanks and drums which have contained such apparently harmless liquids. In these cases there has been doubt whether the explosions were due to the oil itself or to the presence of traces of more volatile combustibles. To remove doubts and to emphasise the need for the application of precautions without exception, the Chief Inspector of Factories initiated an investigation by the D.S.I.R. and the Fire Offices' Committee Joint Fire Research Organisation. This report, 'Explosions in Oil Drums and Tanks,' was recently issued by the Stationery Office (6d.).

A series of carefully controlled experiments with 5-gal. and 19-gal. drums, using kerosene, tractor vaporising oil, diesel oil, decalin, linseed oil, with and without solvent, oleine and arachis oil, showed that the application of heat to the outside of the drum could raise the temperature inside until it was approximately that of the flash point of the oil, and thus cause an explosion. In no case was the flame allowed to touch the vapour. A small relief vent, such as an open filling hole, had no effect on the explosion. Not all the materials tested produced an explosion, but the report points out that this does not mean that they would never do so in ordinary industrial practice. The intensity and extent of heating applied would greatly affect the rise in temperature within a container, and the risk of an explosion. Moreover, the addition of a small proportion of a more volatile oil has a disproportionately large effect in lowering the flash point of the mixture.

The experiments outlined in the report show that explosions are to be expected under the circumstances described due to decomposition of the oil or by formation of oil mists, and that the necessary precautions should be taken in the case of both high and low flash-point liquid containers.

### **£30m. refinery programme completed**

THE new catalytic cracker at Stanlow refinery (Cheshire) is now in commercial production, turning out products at a rate of over 1,000,000 tons a year. Built at a cost, including auxiliaries, of £8,000,000, its completion marks the final stage in Shell's £30,000,000 post-war refinery expansion programme in the U.K. This programme, which has established Shell as the largest refinery operator in the country, controlling an output of 8,500,000 tons p.a. of petroleum products, has included the construction of major extensions both at Stanlow and at Shell Haven in Essex.

The new 'cat cracker' is noteworthy not only because it will increase the supply of petrol but also because it marks another stage in the integration of Shell's nearby chemical plant with the refinery's operations. Now that the new unit is in production, the chemical plant will be able to increase its output of chemical solvents, such as acetone. Moreover, it will result in the recovery of about 10,000 tons p.a. of sulphur by providing the feedstock for a sulphur-recovery plant that is now nearing completion. Ultimately, other by-products from the unit will serve as raw material for the production of tetraethyl lead (an important blending agent for petrol) by the Associated Ethyl Co. at its new plant at Ellesmere Port, Cheshire.

The Stanlow cat-cracker is Shell's second to come into operation in Western Europe. The first was at Rotterdam, which was also the first of its type to operate in this part of the world.

A similar unit is to be erected at Berre-L'Etang, near Marseilles, in the south of France. All three of these plants have the same basic design which involved 1,000,000 man-hours of work on the drawing board.

### **New sulphur winning processes**

DEVELOPMENT of a new process which makes possible the use of immense quantities of brackish or sea water for mining sulphur in tidewater areas has been announced by the Freeport Sulphur Co. A company official said that, as a result, sulphur would be extracted for the first time from underground salt domes at sites where fresh water was not available and the cost of piping it from distant sources would be prohibitive. The process solved the problems of corrosion and scale which hitherto made impossible the use of water having a high salt content. It was expected to stimulate greater interest in prospecting for marshland brimstone and to facilitate the future development of salt domes in the Louisiana and Texas tidelands.

It is reported that the Freeport Co. will use the process to mine a deposit of elemental sulphur at Bay Ste. Elaine, a marshy area near the Gulf of Mexico, 60 miles south-west of New Orleans and 35 miles from the only source of fresh water.

Another interesting development in the winning of sulphur is that operated by the Chemical Construction Co. with which, it is claimed, commercially pure sulphur can be produced from deposits with as low as 20% of sulphur at \$18 a ton, compared with the present price of \$22. The low-grade ore is ground to separate out the free sulphur, then suspended in water and heated to above 120°C. to free the sulphur, which on cooling coalesces into nodules of 98% purity. The nodules are sifted out and the remainder goes through two flotations to yield 95% sulphur. This is added to the sulphur in the melt tank and filtered to yield 99 to 100% pure sulphur.

### Practical help for victims of gassing

**I**NDUSTRIAL gassing accidents, fortunately, are comparatively rare. Because of this and also, to a lesser extent, because of the sometimes unusual nature of the causative agents, there has been delay in exact diagnosis when the victim has been sent to hospital and, therefore, a delay in selecting proper treatment. Furthermore, certain casualties require special treatment, details of which are often not readily available. It was with these considerations in mind that the Works Safety Committee of the Association of British Chemical Manufacturers have considered means of making immediately and easily available a correct history and diagnosis of cases of gassing sent from chemical works. It was thought that if some system could be devised whereby casualties caused by some of the more common gases could have suitable labels or cards sent with them to hospital, setting out details of the first-aid treatment they had already received and suggesting further treatment, many of the difficulties presented by the unusualness of these casualties could be overcome.

The Works Safety Committee, therefore, appointed a panel composed mainly of industrial medical officers to investigate the whole matter and, as a result, a series of labels has been evolved. The Council of the Association has welcomed the scheme and has recommended all members who may at any time have gassing casualties to institute this system of sending a card or label with them to hospital. The booklet, 'Gassing Casualties,' costs 3s. and labels 2s. per dozen of any particular label, and they are available post free from the A.B.C.M., London.

Obviously, this scheme must be introduced carefully in order not to offend the professional susceptibilities of hospital staffs, and it is essential that there should be consultation and discussion with local hospitals beforehand. Obviously, the scheme should not be applied from scratch to the first casualty which occurs.

It is not intended to use the labels as instructions to medical staff who are familiar with the action to be taken, but only to give guidance which a casualty officer, faced with the need for immediate action in an unfamiliar type of emergency, could follow with safety, pending further advice.

The scheme has also been brought to the attention of the Chief Inspector of Factories and the Senior Medical Inspector, who welcome it and support it, feeling that emergencies of this type may be dealt with more speedily and effectively and lives may be saved with the collaboration and transfer of information which is envisaged. The medical inspectors will assist in explaining and facilitating the launching of the scheme. The Ministry of Health have also approved the idea.

### Tin oxide conducting film on glass

**A**METHOD of coating glass with a thin transparent film which will conduct electricity has been developed at the National Physical Laboratory, Teddington. The film can be heated by passing a current through it. It could be used, for example, in aircraft windscreens to keep them free from ice and snow.

The layer on the glass is an extremely thin one of an oxide of a metal. Not all metals produce transparent oxide films. One which has given good results is tin. It can be deposited very evenly on the glass by techniques which are already well understood in industry. The glass is then heated to near the softening point and cooled again. The tin oxidises

and becomes transparent. Finally the film is washed in water and dried, a step which increases its conductivity. After this it is hard and inseparable from the glass surface and it is resistant to chemical attack.

Visibility through the glass is reduced by the metal coating by an amount which is negligible for all practical purposes. The coating has a resistance of about 1,000 ohms between the opposite edges of any square area. Enough current can be passed through it to keep the surface of the glass so hot that it is impossible for ice and snow to form on it or for condensation to make it misty. In practice the film would be sandwiched between two layers of glass.

The treated glass may also be suitable for car windscreens and for shop windows to keep them from steaming over in cold weather.

Ceramic materials as well as glass can be treated by the process, and there are many uses for it in the electrical industry, for instance in the production of fixed and variable resistances. The glass can be used to avoid static charges accumulating on the windows of instruments when they are cleaned in dry weather. This static electricity can lead to false readings and it was to solve this problem that the research was originally begun at the N.P.L.

The process is being patented.

### Fuel and chemicals from Iraq's oil

**G**REAT progress is being made in developing petroleum and chemical industries in Iraq, according to a report issued by a World Bank mission which visited the country recently. Although productivity is low at present, developments are said to have been handicapped by financial stringencies. The report states that Iraq possesses impressive potentialities apart from petroleum.

By 1956, oil revenues are expected to rise to 59,000,000 Iraqi dinars (\$65,200,000) and to a net total of 214,000,000 dinars (\$599,200,000) over the next five years. The mission recommended that these rising revenues should continue to be devoted to development. In fact, the total cost of the development programme recommended is estimated at roughly \$470,000,000 for the period 1952-57 inclusive.

One project for the petroleum industry, which was actually planned five years ago but could not be started at the time owing to world shortages of steel and equipment, is scheduled to be completed this month, eighteen months after it was begun and six months ahead of schedule. This is a 560-mile 30-in. pipeline from Kirkuk, north Iraq, to Baniyas, on the Mediterranean, built for the Iraq Petroleum Co. and designed to convey 14,000,000 tons p.a. of crude oil from Kirkuk. For this project alone, 35 ships were used to bring 160,000 tons of 30- and 32-in. pipe and 23,000 tons of 26-in. pipe from America. Some 50,000,000 ton-miles of rail and motor transport were involved in distributing the pipe from the ports of Tripoli, Lebanon and Basrah, on the Persian Gulf, to site for assembly. One interesting feature of the project was the ordering of equal quantities of 32-in. and 30-in. pipe, which enabled freight space to be halved simply by nesting the smaller pipe inside the larger. Although all the steel for the pipe came from the U.S.A., equipment and materials for the pumping stations along the route of the pipeline came from the U.K. A 532-mile double line of 12-in. and 16-in. pipe already connects Kirkuk with Tripoli.

Apart from petroleum, the largest and most promising possibility in the development of other industries is the establishment of a chemical plant at Kirkuk, the centre of most of Iraq's petroleum production and one of the largest



and richest oilfields in the world. This plant could use natural gas, now almost totally wasted, and gypsum as raw materials, and could produce annually 500,000 tons of ammonium sulphate fertiliser, 100,000 tons of elemental sulphur, 10,000 tons of carbon black and 300,000 tons of cement.

Plans for the establishment of a vegetable-oil-processing plant using Iraqi-grown cotton seed to produce hard oils were briefly described in *INTERNATIONAL CHEMICAL ENGINEERING*, June 1951, p. 292. Such a project would provide raw materials for the existing soap industry, which is based at present on imported hard oils, and for the production of synthetic ghee as a foodstuff for human consumption, which could be produced at a lower cost than the natural ghee at present used.

Further development of the petroleum, chemical and allied industries in Iraq is today not so much dependent on direct financial assistance as on improvement in the standards of living, education and health. If the country's valuable natural resources are to be fully developed and the desired technological level is to be obtained much better education and health facilities must be provided.

### **Chemical industries for Ceylon**

**C**HEMICAL industries which might be introduced into Ceylon were recently described by Mr. L. H. Manderstam, consulting chemical engineer, at a meeting of the Ceylon Association of Sciences in Colombo. Three chemical projects being planned by the Government of Ceylon were noted in *INTERNATIONAL CHEMICAL ENGINEERING* (September 1950, p. 396). Mr. Manderstam referred to these projects, which are for the processing of vegetable oils, manufacture of caustic soda and production of nitrogen fertiliser.

The vegetable oil project, which is part of the Colombo plan for developing S.E. Asia, is mainly concerned with copra and its products, and secondly with certain oil seeds. It is comprehensive, embracing the refining of oils and also the production of fatty alcohols, fatty acids, glycerin, and cattle food. An oil-seed processing industry already exists which, however, is based on rather primitive methods and wastes as much as 8 to 20% of the oil. The caustic soda scheme will be based on the conversion of local salt into caustic soda, hydrogen and chlorine. The hydrogen will be used for the conversion of coconut oil into fatty alcohols for detergent manufacture. The chlorine will be used for the manufacture of DDT, the plant for which is expected to be installed by a United Nations' agency.

Plans for a steel industry have also been approved by the Ceylon Government. We understand they recently called for tenders for the establishment of an iron and steel plant at Enderamulla, about seven miles from Colombo. The plant required includes a unit for the production of 5,000 to 7,000 tons of pig iron p.a., which will eventually be raised to 18,000 to 25,000 tons p.a. A basic electric steel furnace or an oil-fired basic open-hearth furnace is required having a capacity of about 70 tons/day of ingots as well as a rolling plant capable of dealing with 20,000 tons of ingots p.a. Plant and equipment for fabricating the steel into sections, angles, etc., is also to be installed. Hydroelectric power would be made available at the site. (Further details of this project can be obtained from the Commercial Relations and Exports Department of the Board of Trade in London.)

A wealth of valuable chemical products is latent in the jungles of Ceylon, which cover about two-thirds of the island. A wood distillation industry could be set up to

produce chiefly charcoal, acetic acid and wood alcohol. There are several thousands of acres under illik grass, which is very suitable for paper-making. There are also deposits of minerals which could be more intensively exploited. These include graphite, mica and ilmenite. The ilmenite deposits are reported to be among the richest in the world, with a titanium oxide content of about 54% and, in view of the increased interest in titanium metal for high-temperature uses, it should be worth mining.

As mentioned, hydroelectric power is being developed, furthermore, water supplies for industrial purposes are readily available. Ceylon has no local fuel other than wood or peat and full consideration is being given to the gasification of peat for fuel. The development programme of the Colombo Plan, launched in October 1950 by the British Commonwealth Governments, envisages a total expenditure during 1951-57 of 1,359,000,000 rupees for Ceylon, of which 37% will be spent on agriculture, including hydroelectric power, 8% on fuel and power, and 5.5% on industry and mining.

It is clear that in the industrialisation of Ceylon, as in the case of other countries, the help of the chemical engineer is badly needed. The question is, can Britain or the United States, both short of chemical engineers themselves, spare the technologists to design and engineer the projects?

### **Towards better lubricants**

**T**HE increased use of machinery operating through wide ranges of temperature has resulted in attention being paid to the development of lubricating oils with improved viscosity temperature characteristics. In one recent investigation, the polymers and copolymers of vinyl esters of the long-chain fatty acids were tested as viscosity index improvers for lubricating oils, an application likely to be of commercial value, since the monomers can be easily prepared in good yield from inexpensive and readily available raw materials.

Polyvinyl caprylate and palmitate, and copolymers of vinyl palmitate with vinyl acetate have in particular been shown to be effective viscosity index improvers, the improvement caused by the copolymers increasing with increased vinyl acetate content. It was also found that the solubility of the copolymers in lubricating oils decreases as the vinyl acetate content increases, the best results being obtained with a copolymer containing about 25 mole % of vinyl palmitate.

In general, it would seem that the viscosity index improving effect of a polymer or copolymer in a lubricating oil is enhanced as the solubility of the polymer decreases and as its concentration increases. These investigations may have a considerable influence on future developments in this field.

### **'Chemical & Process Engineering'—May**

**The Sindri Fertiliser Factory.** The £17,000,000 fertiliser factory at Sindri, Bihar, India, was officially opened on March 2 by the Prime Minister, Pandit Nehru. Government-owned, it is designed to produce more than 350,000 tons of ammonium sulphate p.a., about one-seventh of India's fertiliser needs. The complete factory was designed by the Chemical Construction Corporation, U.S.A., and construction and procurement of equipment were handled by the Power-Gas Corporation Ltd., England. An illustrated description of the factory will appear in the next issue of *CHEMICAL & PROCESS ENGINEERING*.



# Chemical Engineering Progress in the Gas Industry

By G. U. Hopton, B.A. B.Sc., M.I.Chem.E., M.Inst.Gas E.

(Research Department, North Thames Gas Board)

## PART I. MANUFACTURE AND PURIFICATION OF GAS

The gas industry is a good example of how an industry can keep down costs and raise efficiency by the proper application of advances in chemical engineering. This is shown by the fact that while the cost of its raw materials rose more than fourfold during 1935-48, the industry was able to keep down the prices of its products to less than three times the 1935 level, during which time the value of the output per employee rose from £328 to £613 p.a. These are good reasons for this survey by a leading gas engineer of the present status of chemical engineering in the gas industry. If another reason is needed it is that the gas industry is a very big buyer of plant and equipment; in 1948, for instance, it spent £10,500,000 on plant and machinery out of a total capital expenditure of just over £18,500,000.

THE efficiency of the gas industry depends to a great extent on chemical engineering. The manufacture of gas is a chemical process in which chemical engineering plays a major part; it has been stated that 'the best training for the specialist in fuel technology is a course in chemical engineering followed by a specialist course either in fuel technology or in a subject such as gas engineering.'

The purification of gas comprises cooling and condensation, separation of tar, naphthalene, ammonia and benzole, and removal of sulphur compounds, in plant based on application of the principles of heat transfer, mass transfer, and chemical reaction. The storage and distribution of gas present problems in fluid flow and corrosion. The efficient utilisation of gas depends on knowledge of the principles of gaseous combustion and of heat transfer by convection and radiation under widely different conditions. The working up of by-products, which are important as raw materials in the chemical industry, is carried out in a variety of units such as stills, fractionating columns, centrifuges, driers and contact and chamber acid plant. In this large field of activities the gas industry has made use of advances in chemical engineering to improve existing practice and to introduce new processes.

Research on new processes is actively pursued. For example, the complete gasification of coal is being studied so that use may be made of non-coking coals for gas manufacture. Processes have been developed for the use of fuel oil in making peak load gas, and for the synthesis of methane to enrich gas of low calorific value. Alternative methods are being sought where existing processes for the purification of gas have become undesirable because of high labour requirements or difficulties in disposal of effluents. This research work passes through a pilot-plant stage to the full scale when it is promising, and the development from the laboratory

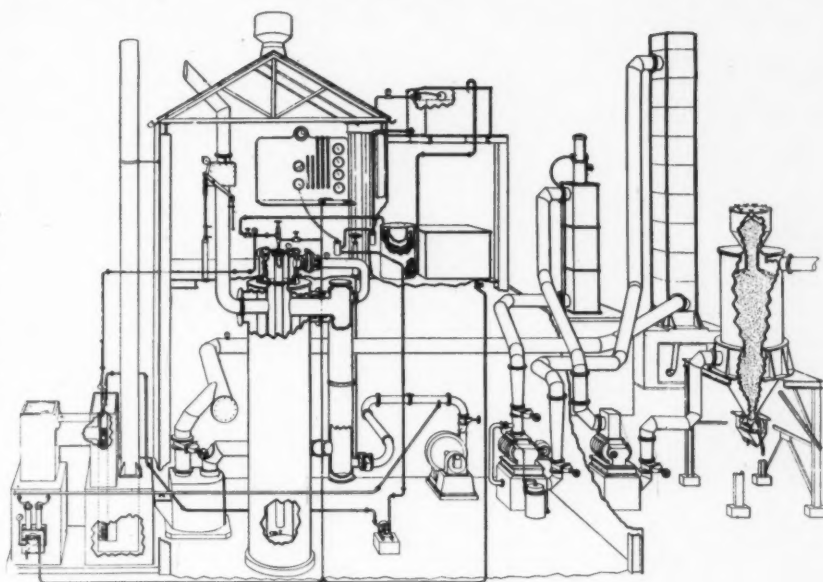


Fig. 1. Diagram of oil gas plant.

requires the services of chemical engineers. It is the purpose of this article to describe some of the ways in which chemical engineering is applied in the gas industry, and to indicate the nature of current problems on which the chemical engineer is engaged.

### Manufacture

Coal gas is manufactured by the carbonisation of coal in horizontal, continuous vertical and intermittent vertical retorts, and in coke ovens. Water gas is manufactured by the gasification of coke with steam. These processes are under the control of the gas engineer, but there are many important features which depend on chemical engineering. One example is the improvement in working conditions in retort houses by the provision of ducts for extracting the air and removing the dust from it by impingement on water in a

Drummond plant.<sup>2</sup> Dust removal from water gas by cyclone separators has been introduced before the carburettor, and dust is also removed from the waste gases leaving the generator and the waste heat boilers. Wet handling of the separated dust in the form of a slurry by sluicing with water is being studied.

The principles of fluid flow are applied to the calculation of pressure drop in the design of producer gas mains so that both sides of a setting may be equally heated. Although the thermal efficiency of gas manufacture is already high, steps are being taken to improve it by a study of the preheating of secondary air for producer gas combustion, and the use of back-pressure turbines as prime movers in water gas plant, and of gas turbines driven by the waste gases from the producers.

The standard of insulation of retort

settings has been improved by the use of high-temperature insulating bricks made from expanded fireclay. By substituting 18 in. of this material for the 6 in. of second-quality firebrick normally used for the exterior of the retort setting walls, it has been possible to reduce the heat losses from a continuous vertical retort bench carbonising 300 tons of coal a day, by an amount equal to a saving of  $\frac{1}{2}$  ton of producer fuel a day. Similarly, by substituting high-temperature insulating brick for the fireclay lining of waste gas flues between the retort setting and the waste heat boiler, the heat losses from the flues have been reduced by an amount equivalent to an increase in waste heat steam production of about 2%. These reductions in heat loss may appear small, but taken throughout the 20 years' life of a retort they represent a significant saving in fuel.

The gas industry has the advantage that gas can be stored in holders to meet hourly and daily fluctuations in demand, but the problem of how best to supply gas for two or three weeks of very cold weather remains. The provision of carbonising and water-gas plant for such peak loads is expensive, and this fact taken in conjunction with the shortage of coal has led to the development of methods of manufacture of gas from fuel oil in relatively simple plant. Fig. 1 is a diagrammatic drawing of a peak-load gas plant using the partial combustion of gas oil, which has been developed to the full scale by M. Maccormac.<sup>3</sup> Gas oil preheated by the combustion of tar produced in the process is injected with air preheated by the hot gas into a generator where partial combustion occurs with cracking of the hydrocarbons, producing a gas of suitable calorific value for distribution, but of high specific gravity and unusual combustion characteristics, so that it must be mixed with a larger volume of coal gas, which is in fact the case at times of very high demand. Fig. 2 is a photograph of a pilot plant developed by H. Stanier and J. B. McKean<sup>4</sup> for the catalytic gasification of petroleum oils. During gas-making, steam and oil are fed separately to the top of the catalyst bed; air is then passed upwards during a 'blow' period to restore the temperature of the bed. This process has the advantage of being applicable to heavy fuel oil as well as gas oil, and the specific gravity and combustion characteristics of the oil gas are normal, but the plant and process are more complicated than that developed by Maccormac.

As an alternative to the use of oil for the enrichment of blue water gas, and as a means of enriching gas produced by the possible future gasification of coke breeze with oxygen and steam, the Gas Research Board has made a study of the catalytic synthesis of methane.<sup>5</sup> Fig. 3 is a photograph of a pilot plant in which the problem of the control of the temperature of the catalyst due to the exothermic nature of the reaction between carbon monoxide and hydrogen was solved by the re-circulation

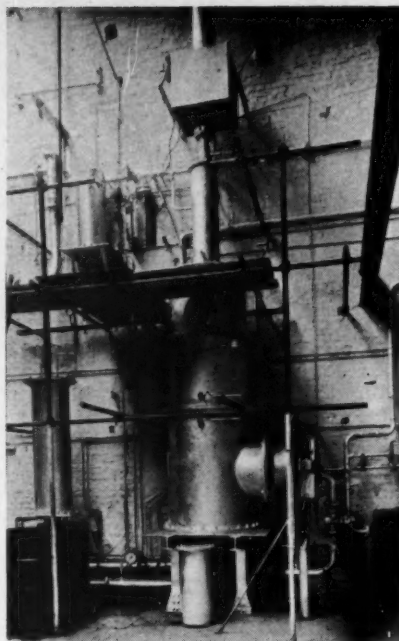


Fig. 2. Pilot plant for catalytic oil gas.

of gas. The plant consists of a circulating fan, a catalyst vessel and a cooler, connected in closed circuit with provision for addition of reactants and withdrawal of products. The Gas Research Board has carried out experiments on the complete gasification of coal,<sup>6</sup> and has also considered the application of fluidisation and vortex gasification. While these projects are yet in an early stage, it is probable that they will be carried to a larger scale and will require chemical engineering of a high standard to solve the problems likely to arise in working at high temperature under pressure, in designing gas washing plant and heat transfer equipment, and in pro-

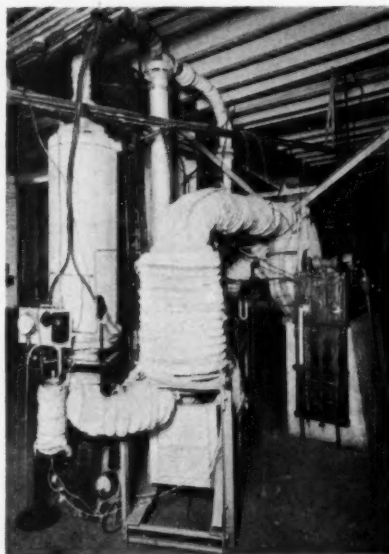


Fig. 3. Pilot plant for methane synthesis.

viding apparatus of a kind not found in conventional gas manufacture.

## Purification

The first requirement in the purification of gas is a supply of cold water. The performance of water cooling towers has been studied by two workers in the gas industry, W. K. Hutchison and E. Spivey.<sup>7</sup> After making tests on commercial cooling towers they designed an experimental cooling tower with two main features, namely close grid packing and trough distributors. Fig. 4 gives a comparison of the mass transfer per unit volume, from which it is seen that the experimental unit is by far the more efficient. The design has been developed to the full scale, and is widely used in the gas industry and elsewhere. Fig. 5 is a drawing of a forced draught cooling tower with trough distributors and grid packing.

When crude gas leaving the retort, coke oven or generator is cooled, condensation of water takes place as well as separation of naphthalene and tar. The problem consists in an analysis of the mechanism of cooling with aqueous condensation, and the design of plant to prevent blockage from naphthalene and tar. There has been a tendency in the past to simplify the theoretical aspect by considering the condensation to take place first, followed by the cooling, making two separate calculations and then adding the required surface areas. In fact, the two processes of heat transfer and mass transfer occur simultaneously. The rate of mass transfer of water determines the rate of transfer of latent heat, which together with the rate of transfer of sensible heat makes up the total heat transfer. It has been shown by L. Silver<sup>8</sup> that although the total rate of heat transfer increases rapidly with rising temperature, the rate of transfer of sensible heat alone is very nearly constant over the whole temperature range in a gas condenser, and it may be used to calculate the size of condenser required.

Four main types of gas cooler, or condenser as it is commonly called, are used in the gas industry. It is a mistake to assume that the atmospheric cooler, consisting of a number of long almost horizontal pipes lying vertically above one another and connected at alternate ends by return bends, is inefficient. When the surface of the pipes is irrigated with water by properly designed trough distributors the gas is cooled to within two or three degrees of atmospheric temperature and considerably below the temperature of the cooling water. A further advantage is that by re-circulation of the water, the necessity for a water cooling tower is avoided. The atmospheric condenser, however, is expensive and occupies much space. The horizontal-tube battery condenser described by H. Hollings and W. K. Hutchison<sup>9</sup> has been widely used. The gas enters at the top of the condenser and flows down one side of a vertical chamber through nests of nearly horizontal steel

tubes which are expanded into fixed tube plates and, crossing at the bottom, passes up to the outlet through similar nests of tubes on the other side of a central division plate. The water flows in series through all the nests of tubes in general direction countercurrent to the gas, and passes from one nest to the next by way of suitably shaped header plates. Recently the trend has been to instal the multipass vertical water-tube condenser in which, by the fitting of vertical division plates in the gas space with gas ways alternately at the bottom and top of the plates, together with corresponding division plates and water ways in the upper and lower water chambers, the two fluids are constrained to make from four to twelve passes up and down the vessel countercurrent to one another. Fig. 6 is a photograph of a Whessoe condenser of this type.

In tubed condensers the efficiency is impaired if naphthalene is allowed to accumulate on the cooling surfaces. Therefore, provision is made for periodic reversal of direction of flow so that the cold end of the condenser is heated to allow a semi-solid deposit to drain away. Flushing with tar may also be practised. An alternative design which overcomes deposition of naphthalene is the direct cooler, known as the washer-cooler. This consists of a scrubber packed with wooden grids up which the gas passes in direct contact with cooled liquor which is recirculated over the packing from a carefully designed distribution system placed over the boards. Hot liquor flows from the bottom of the packing into a settling tank, from the middle of which it is drawn off and pumped through rack coolers irrigated with water. Tar is run off from the bottom of the

settling tank, while the liquor condensed from the gas overflows to a receiving tank. A technical disadvantage of the washer-cooler is that the transfer of heat from gas to cooling water is done in two stages, so that a very close approach in temperature is impracticable.

It is realised that heat is lost in the gas condenser, but it is difficult to recover this heat when the hot gas temperature is only 140 to 180°F. The use of gas condensers for preheating boiler feed water is, however, frequently adopted.

After being cooled, the gas passes through washing plant, and some variation in relative order of the units is practised. Fig. 7, taken from a paper by S. Pexton,

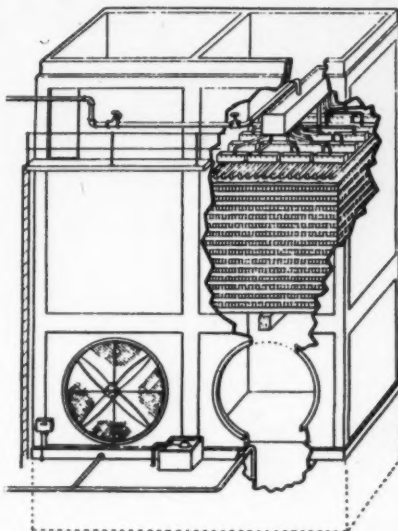


Fig. 5. Diagram of water cooling tower.

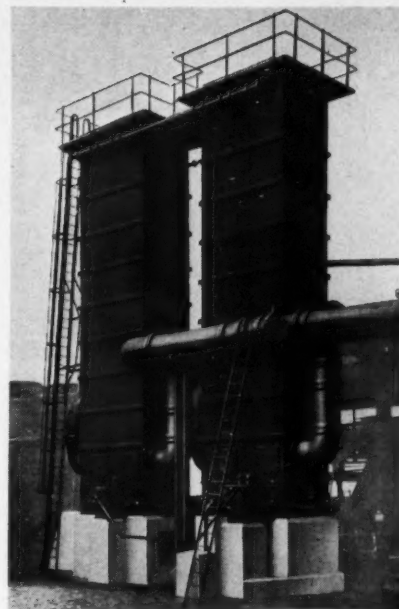


Fig. 6. Vertical water-tube condenser.

G. Dougill and L. A. Ravald,<sup>10</sup> shows the arrangement of washing plant in four recent installations. In scheme (a) the gas is cooled in a primary condenser of the vertical water-tube reversible type, and tar fog is removed after the exhauster by an electrostatic precipitator, after which the gas passes through a naphthalene washer before being cooled in a secondary condenser. This is followed by a second electro-detarrer which protects the ammonia washer from tar fog formed during secondary cooling. In scheme (b) the primary condenser is a washer-cooler. In scheme (c) the primary cooling is done by a horizontal water-tube condenser followed by a water-tube condenser, the pair being reversible; only one electro-detarrer is installed, its position being before the ammonia washer and after the naphthalene washer and washer-cooler. In scheme (d) the relative order of primary condenser, exhauster, naphthalene washer, secondary condenser, electro-detarrer and ammonia washer is the same as in (c), but different types of condenser are used.

The gas industry has sometimes been criticised for not employing tower scrubbers for naphthalene removal and ammonia recovery. This criticism is unfounded in view of the low circulation rate, which would be inadequate to wet the packing. It is necessary to recirculate the washing liquid (gas oil for naphthalene, and water or weak ammoniacal condensate for ammonia) over the packing at several times the throughput rate. For this purpose the static washer (Fig. 8) in which the gas flows in series through a number of sections (or bays) packed with metal or wooden grids is very suitable. The washing liquid enters at the gas outlet end and is recirculated at a higher rate over the bay packing, over-

## CHIMNEY AND FORCED DRAUGHT COOLING TOWERS

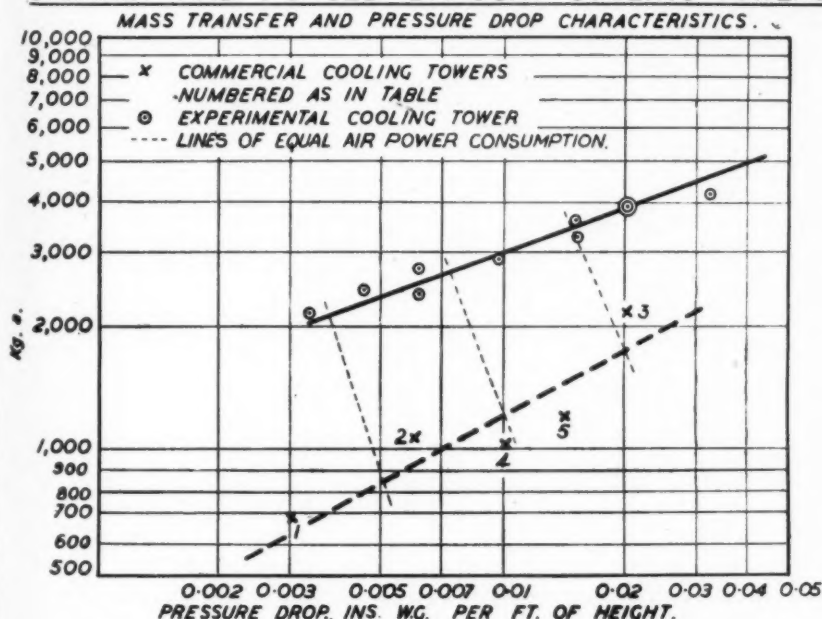


Fig. 4. Graph of performance coefficients for water cooling towers.



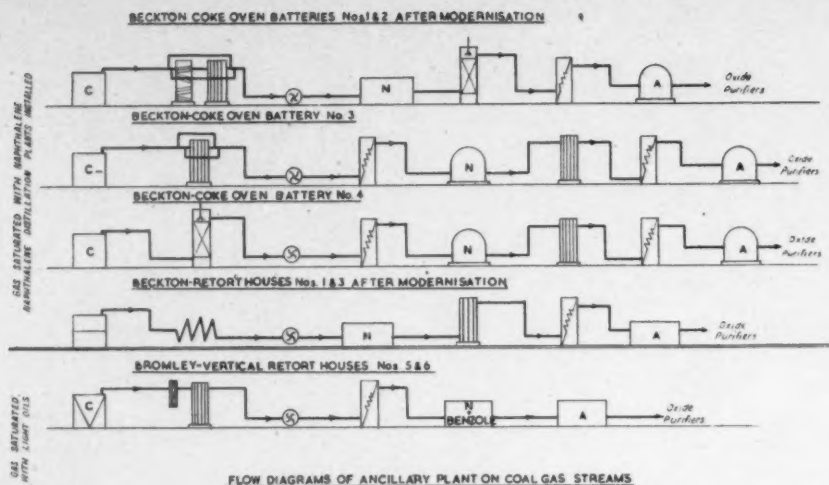


Fig. 7. Flow diagrams of purification sequence.

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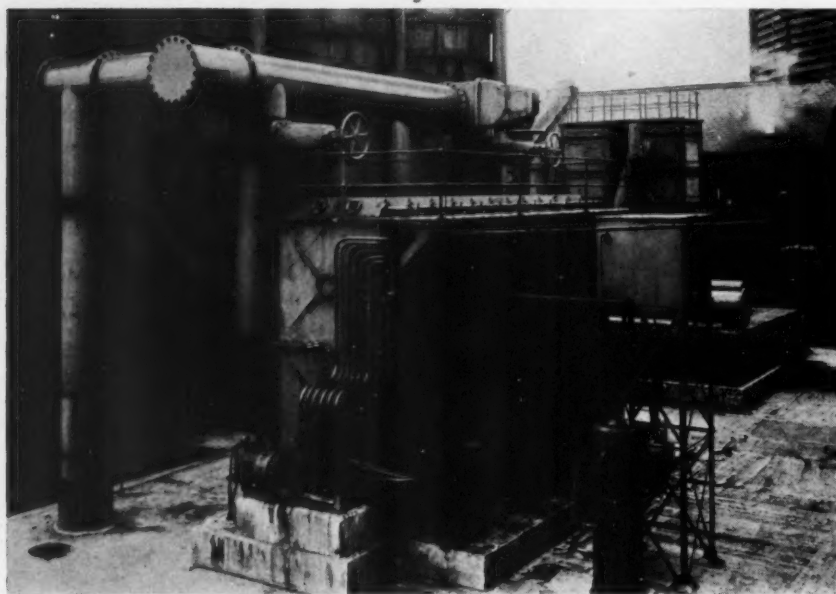


Fig. 8. Static washer.

flowing from bay to bay. Careful design of the distributors above the packing has resulted in very high efficiencies, so that, for example, 99% of the ammonia in the gas can be removed without excessive use of water.

The design of washers has been placed on a sound basis by the fundamental studies of L. Silver,<sup>11</sup> who determined the mass transfer coefficients for the removal of several constituents from gas, so that data are available for the design of washing plant from first principles. The efficiency of washers of different types is tested at every opportunity. The correlation of the mass transfer coefficient with the nature of the constituent and with the properties and flow rates of gas and liquid is continually checked, and improvements in design are studied.

Removal of hydrogen sulphide is carried out by law to an extremely low limit, the test employed being sensitive to  $1\frac{1}{2}$  parts per million by volume. This difficult purification has been carried out for a hundred years by means of solid iron oxide, which has the disadvantage of requiring very large vessels occupying much ground space and of making intermittent demands for heavy manual labour of an unpleasant kind. The replacement of the oxide purifier is a current problem calling for all the resources of chemical engineering. Very many processes using liquid reagents have been proposed, but few have survived to a full scale. Interest is now directed towards two processes with quite different characteristics. The Koppers (U.S.A.) vacuum carbonate process uses a solution of sodium carbonate,

which is circulated over a packed scrubber in which it absorbs hydrogen sulphide, hydrogen cyanide and some carbon dioxide from the gas. The foul solution is then stripped with steam in an 'actifier' operated at an absolute pressure of 4 in. of mercury, the stripped liquor being cooled and returned to the scrubber. The vapours leaving the actifier are cooled, and the hydrogen cyanide is recovered from the mixture of hydrogen sulphide and carbon dioxide before passage to a contact plant for production of sulphuric acid. The removal of hydrogen sulphide from the foul liquor by steam is a physical process which does not proceed to completion, so that the vapour pressure of hydrogen sulphide in the stripped liquor is appreciable. For this reason the removal of hydrogen sulphide from the gas does not much exceed 90%, and other means such as highly active iron oxide in special vessels must be used to remove the remaining hydrogen sulphide. The process, however, has the great advantage of causing no serious problems of effluent disposal. Fig. 9 is a photograph of the Seaboard plant treating 48,000,000 cu. ft. of coke oven gas a day.

The Manchester liquid purification process<sup>12</sup> is fundamentally different in that the foul solution is treated with air, so that regeneration is chemical and can be taken to completion. Hydrogen sulphide removal, therefore, can be carried out to the legal limit, as in the Liverpool plant (Fig. 10). The gas is washed with a suspension of ferric hydroxide in soda solution, using six washers in series. The foul liquor passes to delay tanks in which the hydrosulphide in solution reacts with ferric hydroxide to form ferric sulphide, which is then oxidised to ferric hydroxide and elemental sulphur by the introduction of air through diffusers in four towers

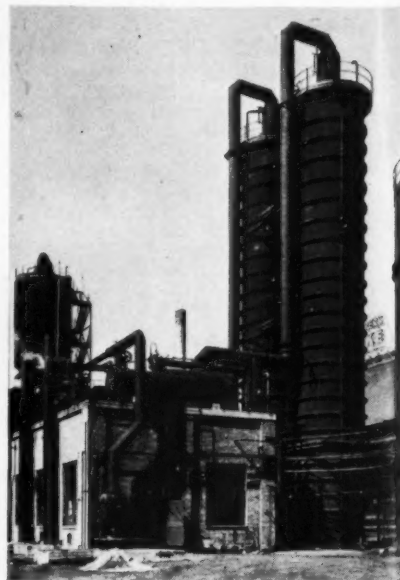


Fig. 9. Koppers' vacuum carbonate plant.

known as oxidisers. The oxidised suspension is returned to the washers. A sulphur sludge containing liquor and ferric hydroxide is withdrawn as product. One of the major problems of the process is the treatment of this material, and vacuum filtration, autoclaving, film drying and dewatering with trough drying are being studied. Another serious problem is the disposal of thiosulphates and thiocyanates which accumulate in the liquor.

Benzole is usually recovered from coal gas after removal of hydrogen sulphide, although the present coal shortage makes the process less attractive than formerly. Two methods of benzole recovery are used, namely active carbon and oil washing. In the first, the gas is passed through several adsorbers containing a bed of active carbon in which are disposed coils through which cooling water is passed to remove the heat of adsorption of benzole. When slip of benzole begins to occur, the adsorber is shut out from the gas stream, and the adsorbed benzole is driven off by live steam, while heating steam is passed through the coils. The design of the plant aims at providing sufficient adsorbers for benzole removal from the gas while one or two adsorbers are being steamed, and of arranging the cycle so that the demand for steam is as continuous and even as possible. A large plant of this type has been described by H. Hollings and S. Hay.<sup>13</sup> Serious difficulties were encountered owing to internal corrosion of the coils, and in a more recent plant the coils are heated during the stripping period by hot water (steam condensate) at 130°C. under pressure in a closed circuit. The same water is then used for cooling during the adsorption period. The equipment consists of a buffer tank into the top of which hot water from the coils is displaced when cold water is pumped from the bottom of the buffer tank into the coils at the end of the stripping period, cold and hot water pumps operating at 50 lb. gauge pressure, an expansion chamber for blowing off dissolved air, and steam heaters and rack

coolers in closed circuit. By this means the corrosion due to the presence of oxygen in the steam and of salts in the cooling water has been avoided with a great saving in the costs of replacement of coils.

The second method of benzole recovery is the oil washing process. The use of gas oil to absorb benzole from coal gas, followed by stripping the benzole from the oil by live steam and recirculating the oil, was introduced on a considerable scale during the first world war. The oil washing rate is about 10 gal. per 1,000 cu. ft. of gas. Determination of the partition factor for carbon disulphide in gas oil showed that a substantial reduction in the organic sulphur content of gas (which gives rise to sulphur dioxide on combustion and is therefore objectionable) would be achieved if the oil rate were increased to 30 gal. per 1,000 cu. ft. The problem was to use

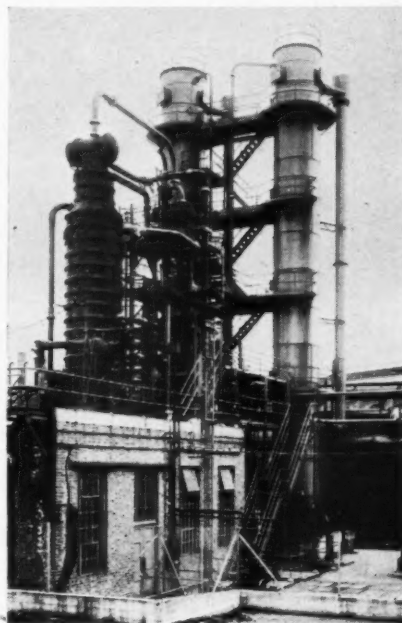


Fig. 11. Benzole recovery plant.

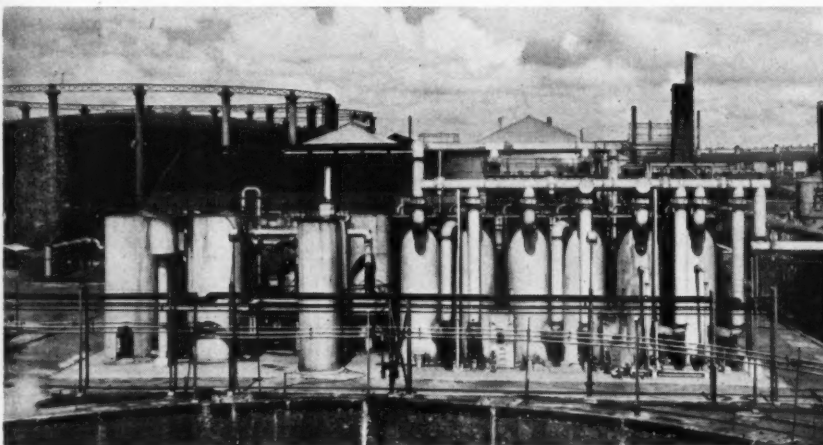


Fig. 10. 'Manchester' liquid purification plant.

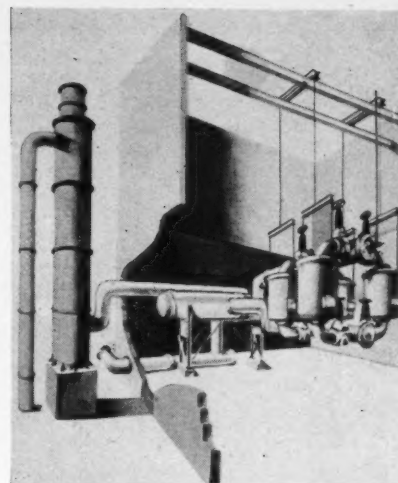


Fig. 12. Diagram of catalytic plant for sulphur removal from gas.

this relatively high oil circulation without excessive steam requirement, and it was solved by stripping at 90°C. under a vacuum of 20 in. of mercury. The use of this vacuum reduced the stripping steam consumption to one-third of that at atmospheric pressure, whereas the steam required to operate the vacuum pump drawing the steam, benzole vapour and permanent gases (dissolved from the coal gas by the wash oil) through a condenser is relatively small in amount. Heat exchange between hot stripped oil and cold rich oil, and between vapours leaving the still and rich oil, is practised, and a careful steam balance is achieved so that all the steam required in the oil heater and for stripping is supplied at 5 lb./sq.in. gauge as the exhaust from the steam turbines used to drive the oil, water and vacuum pumps. Fig. 11 is a photograph of the first plant of this type, which has been described by W. K. Hutchison.<sup>14</sup> Several plants are in operation, including two large installations in Australia. The theory of the oil washing process with design data for the scrubber and the still has been described by L. Silver and G. U. Hopton.<sup>15</sup>

Both the active carbon and the oil washing processes for benzole recovery are capable of reducing the organic sulphur compounds in coal gas from 30 to 40 gr./100 cu.ft. to 10 by virtue of the substantial removal of carbon disulphide and thiophen, but carbon oxysulphide remains in the gas. It can be destroyed by catalytic oxidation, when gas containing less than 3 gr./100 cu.ft. is produced, suitable for flue-less appliances and for special furnace atmospheres. Extensive laboratory research led to the use as catalyst of nickel subsulphide on china clay pellets at 360°C. and 1,000 space volumes per hour. The full-scale plant (Fig. 12) contains four vessels each 4 ft. 6 in. high and 3 ft. in diameter. Coal gas at the rate of 1,500,000 cu.ft./day is preheated in a horizontal heat exchanger, flows through the four catalyst

vessels in parallel, and passes through the heat exchanger to a washer-cooler for removal of the sulphur dioxide by means of cooled recirculated sodium carbonate solution. The plant has been described by R. H. Griffith and J. H. G. Plant.<sup>16</sup> The maintenance of the correct operating temperature in the catalyst bed is essential, and it is accomplished by adding a controlled quantity of air to the gas entering the plant so that reaction between oxygen and some of the hydrogen present in the coal gas supplies the necessary heat. R. H. Griffith<sup>17</sup> has recently described the application of a magnetic oxygen recorder for this purpose. In this interesting device the sample of coal gas after addition of air is passed upwards through both sides of a circular annulus provided with a horizontal connecting tube through which gas is drawn by a magnetic field at a rate dependent on the concentration of oxygen. The gas stream passing through the connecting tube cools a surrounding winding so as to unbalance a Wheatstone bridge and produce an e.m.f. proportional to the oxygen concentration of the gas. This e.m.f. is used to record the oxygen concentration and to control the admission of air to the plant.

**In part 2, Mr. Hopton will deal with the storage and utilisation of gas and the working up of by-products.**

#### ACKNOWLEDGMENTS

Fig. 2, Institution of Gas Engineers; Fig. 3, Gas Research Board; Fig. 4, Institution of Chemical Engineers; Fig. 5, Gas; Fig. 6, Whessoe Ltd.; Figs. 7 and 10, Gas World; Fig. 11, Institution of Gas Engineers.

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## Dust and Disease in Steel Foundries

**M**ETHODS of preventing the production or the inhalation of dust and the possibility of reducing the use of materials containing free silica in steel foundries have been investigated in the U.K. during recent years by a committee representing employers, trade unions and the Government. In 1944 the committee published the 'First Report on Dust in Steel Foundries'; now the second report has been published by the Ministry of Labour (H.M.S.O., 3s. 6d.).

A wide medical survey of the incidence of pulmonary dust diseases in foundry workers is described. Over 3,000 people were examined clinically and by x-ray in 19 foundries. Information about the occurrence of silicosis and other forms of pneumoconiosis was also drawn from more than 60 foundries in different parts of the country. It appears that there is a greater risk of silicosis developing in steel foundry workers with the exception of labourers, than in workers in iron, or mixed iron and steel foundry workers. Workers in steel fettling shops are liable to develop more severe x-ray abnormalities than workers in any other foundry occupation. In fact, the numbers of steel fettlers certified by the Silicosis Medical Board as having silicosis have increased yearly since 1931, and are still increasing. Probable reasons for the increase are given: (1) that the dust from

the process is still not controlled; (2) that many more fettlers are using pneumatic tools; and (3) the output of steel castings has doubled. In contrast, the numbers of cases of silicosis among sand and shot blasters have not shown a comparable yearly increase, probably because the process of blasting is under regulations which prescribe dust control and protection of the workers.

It is pointed out that steel moulders and coremakers, provided that they are not exposed to the inhalation of dust from other foundry processes have a lower incidence of x-ray abnormalities and silicosis than do the steel fettlers. They are exposed to a greater risk of silicosis if silica flour is used in the moulding mixtures without adequate dust control measures.

Welders and oxyacetylene cutters develop siderosis as the result of the inhalation of iron oxide fume, but when welding, oxyacetylene flame cutting or burning are carried on in a steel fettling shop, the welders and cutters are exposed to the risk of silicosis and mixed dust pneumoconiosis. Pure siderosis is not a disabling condition. In common with most other workers in steel foundries, knockout men, sand mixers and crane drivers show a higher incidence of x-ray abnormalities than the comparable group in iron or mixed iron and steel foundries. Men

exposed to the inhalation of dust from the knockout are liable to contract silicosis, but not to the same extent or as rapidly as blasters or fettlers. The risk to crane drivers is apparently greater in the fettling shop than it is in the moulding shop. Furnace workers, including casters, ladlemen, pourers and annealers, develop abnormal x-ray appearances after some years' exposure to the dust and fume arising from their jobs. The abnormal changes are more pronounced in steel foundry workers than they are in iron or mixed iron and steel foundries. The incidence of active tuberculosis among foundry workers is much the same as that found in the general population (3.6 cases per 1,000), though it is recognised that workers already suffering from silicosis are peculiarly liable to contract tuberculosis as well.

It is suggested that steel foundrymen should undergo pre-employment and periodical clinical and x-ray examinations. These have been in force for some years in Scandinavian countries such as Sweden and Denmark, and also in some parts of the United States. The committee spent some time in trying to set up with the British Steel Founders' Association and the British Red Cross Society a comprehensive and practical scheme of periodical medical examinations which would assist further research and could be carried out at the same time as environmental studies. Although it was not possible to set up the scheme, the British Steel Founders' Association have proceeded with a scheme of their own, and a portable x-ray unit is, at present, touring the country so that member firms of the Association may arrange for x-ray photographs on behalf of their employees.

**Methyl chloride precautions.** Information on the safe handling and use of methyl chloride is given in 'Chemical Safety Data Sheet SD-40,' published by the Manufacturing Chemists' Association Inc., U.S.A. When used as a chemical reagent or solvent, methyl chloride presents a variety of hazards in its handling. Although thermally stable, it is subject to slow decomposition upon contact with moisture. At high temperatures it may break down to produce hydrogen, phosphine, chlorine and carbon monoxide. These are all toxic, either alone or in a mixture. Furthermore, it is inflammable. The bulletin sets forth safety rules and precautions to avoid all of these hazards. It also includes regulations which apply to tank vehicles and cylinders used in the transport of the material. These cover loading, unloading and return of empty containers, as well as correct labelling. Effective employee education is stressed and complete information of first aid and medical treatment is given for guidance in the control of health hazards likely to arise where methyl chloride vapours are present.



# CATALYSIS

## Catalytic cracking and polymerisation processes; fused salt systems; acid-base catalysts

By S. L. Martin, M.Sc., A.R.C.S., D.I.C., F.R.I.C.

**F**OLLOWING previous practice, the present report first covers points of general interest such as recent significant advances in the field of catalysis, and then in greater detail, one or two aspects of the subject which either represent new approaches or have been clarified by recent work. A general review of the newer concepts of acid-base catalysis and free-radical mechanisms in relation to polymerisation processes was given in the last report<sup>1</sup>, and before that the interpretation of the action of certain catalytic cracking catalysts as 'acid catalysis' leading to formation of carbonium ions was briefly discussed.<sup>2</sup> These two topics, which have certain points in common, have continued to receive considerable attention, and the recent literature provides sufficient data to substantiate what were previously only concepts or working hypotheses. However, before continuing our review of them, some recent work on catalysis in fused salt systems will be described briefly, as representing a novel approach.

### Petroleum cracking

Catalytic cracking of petroleum products and polymerisation processes continue to vie with each other for first place of interest in the literature; the Fischer-Tropsch and related syntheses, esterification, hydrogenation and alkylation catalysis have also received concerted attention.

Sittig<sup>3</sup> has reviewed in a series of four articles the six main cracking processes—Houdry, Thermoform, McAlfee, fluid, suspensoid, and cycloversion. In the 4th Unit Processes Review of the American Chemical Society,<sup>4</sup> Haensel and Sterba<sup>28</sup> dealing with catalytic decomposition of hydrocarbons, refer to two new concerns to manufacture cracking catalysts, one in Salt Lake City aiming at 50,000 tons per year from Utah clays, another in Chicago at 3,000 tons per month of synthetic material. A report by Johnson, also mentioned, indicates catalyst consumption (loss) as 300 tons per year in a plant which ran continuously for 677 days, or 0.212 lb./barrel of oil processed. Thornton<sup>5</sup> has described a new chrome synthetic catalyst containing 0.003% Cr, which in bead form in the Thermoform process has the usual cracking activity, but reduces afterburning in the regeneration zone through

the promotion of carbon dioxide-monoxide ratios higher than those which occur with conventional catalysts. It is reported<sup>6</sup> that the Shell Group have now completed the first modern cracking unit in Europe, located near Rotterdam; this, a part of their post-war refinery expansion programme which includes two large projects in England, will have a full capacity of 1,250,000 tons p.a.

Butler and Bennister<sup>7</sup> have surveyed the U.K. petroleum industry. Pointing out that petroleum products provide raw material for 25% of the total U.S. synthetic organic chemical production (largely through natural gas and reactive hydrocarbons obtained as by-products in refinery processes), they indicate how a similar development in the U.K. will be fostered by the three major oil refinery projects which together aim at increasing refinery output from 5,000,000 tons p.a. in 1949 to 19,000,000 tons in 1953.

### Esterification

For esterification processes, of such importance in the production of plasticisers and alkyds, Reid<sup>4</sup> considers that the ultimate in catalysts appears to be trifluoroacetic anhydride which, with alcohols and fatty acids, causes spontaneous esterification with the evolution of sufficient heat to ensure that the reaction is complete before the mass cools entirely. In the case of high temperature esterifications, he suggests that the main value of the catalysts used ( $\text{Ca}(\text{OH})_2$ ,  $\text{PbO}$ ,  $\text{ZnO}$ , clays, etc.) is in increasing the rates of reaction.

### Nitriles

'Acidic' oxide catalysts are generally used in the formation of nitriles by reacting aromatic hydrocarbons with ammonia, temperatures in excess of 900° F. being usually required. Klinitas and Rasmussen claim<sup>8</sup> that by mixing 1% water vapour with, for example, toluene and ammonia, and catalysing the reaction over a molybdenic oxide-alumina catalyst by an adiabatic rather than an isothermal process, an increase in yield of some 20% is obtained; also, by using an activated carbon as catalyst and mixing about 15% air with the toluene/ammonia stream, equivalent yields are obtained at some 200-300° F. lower than with the oxide catalyst. For the

production of dimethylaniline, Evans and Bours<sup>9</sup> have reported that the vapour phase reaction over an activated alumina catalyst is best, and that aniline with 5 moles dimethylether at 285° C. gives an almost quantitative yield of a 99% pure product in a pilot plant capable of 4-5 lb./hr. output.

### Dehydrogenation

For dehydrogenating *n*-butenes to 1,3-butadienes, a new calcium nickel phosphate catalyst has been reported,<sup>10</sup> capable of giving 93-97% ultimate yields at 20-45% conversion levels in the laboratory, and 86-88% yield at 35% butene conversion in the plant. This is considered particularly useful in industry when utilisation of *n*-butene is necessary, comparing with previous ultimate yields of 70-85% at 40-20% conversion levels. The catalyst contains 55.77, 31.31, 5.22, and 6.24 wt. % respectively of phosphate, calcium, nickel and water, and is prepared by co-precipitation from mixed chloride solutions. Promotion with chromium trioxide increases stability and activity, and prolongs life in use at 550-650° C. The catalyst, which is a single-phase solid solution, is susceptible to poisoning by metallic oxides of an alkaline nature, and to nickel-containing steels which lower the selectivity, but not to ferric oxide.

### Atomised powders

For many purposes, such as certain hydrogenation, de-hydrogenation, polymerisation and isomerisation processes, metals or alloys used as catalysts are required to be in a high state of dispersion. Some results with atomised powders, which appear promising, have been described recently.<sup>11</sup> Pure metal wires, or twists of wires of more than one metal where alloys are required, are atomised with a pistol using oxygen and acetylene for melting and compressed air for spraying, and sprayed parallel to the surface of a non-inflammable liquid or into a large cylinder with a sieve, in such a way that the particles cool before contacting each other. Thin porous screens, serviceable for continuous contact processes, can be obtained by spraying on to glass surfaces.

The procedure allows a wide range of alloys of varying compositions to be ob-

tained readily in powder form as particles 0.002-0.08 mm. in diameter. The particles should be dried in an atmosphere free from poisons. Powders of metals such as nickel and its alloys which have become deactivated by superficial oxidation, can be re-activated by relatively mild treatment with a 5% solution of sodium hypochlorite in the presence of acid, followed by filtering, washing, and reduction in hydrogen at about 250° C. They should be kept in a carbon dioxide atmosphere.

### New literature

The following recent publications are worthy of note. In their book on the Fischer-Tropsch and related syntheses, Storch, Golumbic and Anderson<sup>12</sup> also summarise recent advances in theoretical and applied contact catalysis; the recent work of the U.S. Bureau of Mines Group is described, and detailed accounts are given of the preparation, properties, and structure of the various catalysts.

Of particular interest in the third volume of 'Advances in Catalysis',<sup>13</sup> published in 1951, are the articles on 'Catalytic Cracking of Pure Hydrocarbons,' by V. Haensel, 'Chemical Characteristics and Structure of Cracking Catalysts,' by Oblad, Milliken and Mills, and, on the more academic side, 'The Poisoning of Metal Catalysts,' by Prof. Maxted, and 'Magnetism and the Structure of Catalytically Active Solids,' by Prof. Selwood.

The 39 papers presented at the International Colloquium on Adsorption and Heterogeneous Kinetics, held in Lyons in 1949, have now been published,<sup>14</sup> as have been certain of those read at the Symposium on Polymer Chemistry (Plastics) held in England in 1950.<sup>14, 22</sup> The increasing attention being given to the problems of the fluidisation process is reflected in the titles of the papers due to be presented at the forthcoming conference on 'Fluidisation Technology,' to be held in London from June 11 to 13, 1952, as a joint meeting of the Chemical Engineering Group of the Society of Chemical Industry and the Royal Institute of Engineers of Holland. Of the eleven scheduled papers, four deal specifically with catalytic processes and four with general fundamental aspects of fluidisation.

### Catalysts comprising fused salt systems<sup>15</sup>

In many catalytic processes involving the use of a Friedel-Crafts type of catalyst, the necessity of using a solvent medium is a disadvantage. It is not altogether surprising, therefore, to find early suggestions for the use of molten salts as catalysts through which the vaporised reactants are bubbled. In such cases the melts also effectively replace the organic solvents. The patent literature contains many references to the use of materials such as AlCl<sub>3</sub> in diluents such as SbCl<sub>3</sub> or alkali halides for hydrocarbon synthesis; to halogenations of benzenes, acetylenes and ethers using

melts of halides of metals such as Fe, Al, or Cu, with or without the addition of alkali metal halides to form lower-melting eutectics or to modify the activity; and to the use of similar melts for acetylene polymerisation, which has also been investigated by Swan and co-workers between 1946 to 1949.<sup>16</sup>

Norman and Johnstone have pointed out<sup>15</sup> that the inorganic salts previously used have all been of the co-valent type, that is, they exist in the solid state largely as molecules held together by molecular forces to form 'molecular crystals,' the atoms of the constituent molecules being bound by co-valent forces. This distinguishes them from salts, such as the alkali metal salts, which exist as 'ionic crystals,' comprising largely separate ions of metal and acid radicals, held together by electrostatic forces. (The ionic salts used above in conjunction with the co-valent salts served primarily to give lower-melting eutectics, to stabilise the melt, or to modify its catalytic activity.) Very few inorganic salts show *entirely* co-valent or ionic properties, and many show approximately intermediate behaviour. Examples are:—

Largely co-valent: BeCl<sub>2</sub>, ZnCl<sub>2</sub>, HgCl<sub>2</sub>, halides of B, Al, Ge, C, Si, Ti, and Sn.

Largely ionic: Halides, carbonates, sulphates, tungstates, etc., of Li, Na, K, Cs; Ca, Ba, Sr halides, Hg<sub>2</sub>Cl<sub>2</sub>, CdCl<sub>2</sub>, PbCl<sub>2</sub>, PbBr<sub>2</sub>.

Partially ionic: Cu & Ag halides, MgCl<sub>2</sub>, PbCl<sub>2</sub>, ThCl<sub>4</sub>, FeCl<sub>3</sub>, MnCl<sub>2</sub>.

### Catalysis by melts of ionic salts

Norman and Johnstone concluded that from our rather meagre knowledge of the structures of such salts in the molten state, salts of those metals which are known to be catalytically active should themselves be catalysts in the molten state, which contains relatively 'free' metal ions. The probability of migration of these metal ions to, or on, the surface and within the melt is quite high, so that a continuous source would be available for catalysing reactant molecules. Thus the seriousness of the type of surface poisoning common to many metal catalysts would be lessened, especially since the adsorbed poison molecules would themselves have a high mobility on the surface of the melt.

The criteria deciding choice of suitable salts are: The m.pt. must be in a suitable temperature range, preferably below 500° C., which is an average temperature for metal catalysts; the salt must be stable at the m.pt. and should have a relatively low vapour pressure in the melt; finally, the salt should undergo no changes in composition, for example, by reaction with reactants or products, but the metal ion should be capable of complex formation with at least one reactant. Application of these criteria leaves a limited list of salts likely to function by themselves, though

others may in binary or ternary melts.

Norman and Johnstone record experiments which support these ideas. In one series, acetylene was bubbled through the melt and appearance of carbon in it used as a criterion of catalytic activity for acetylene decomposition. The molten chlorides of Cu, Mn, Ni and Fe (all metals themselves known to be catalytically active in this respect) were active; those of Cd, Sn and Pb (metals themselves inactive) were not active. The second series concerned the dehydrogenation of ethyl alcohol for which the metals copper and silver are known catalysts. On passing ethyl alcohol vapour through molten cuprous chloride, evidence of a mixed action—decomposition and dehydrogenation—was obtained. Decomposition was ascribed to the action of cuprous chloride itself, and dehydrogenation to that of the copper ions. In support of this, it was found that a solution of metallic silver dissolved in the molten silver chloride increased the relative amount of dehydrogenation which occurs. Finally, these authors reported that so far no melts had been found which would catalyse the CO/H<sub>2</sub> synthesis.

### Acid-base type of catalysts

Some very recent publications have contributed materially to the overall understanding of the action of these catalysts, and it is now possible to see signs of some basic principles common to catalytic action in such apparently diverse processes as oxidation by oxides such as cuprous oxide, polymerisation by halides such as aluminium chloride or boron trifluoride, olefin esterification by halides, and hydrocarbon cracking over oxides such as aluminium oxide. Plesch<sup>17</sup> is of the opinion that at least for polymerisation of olefins the cationic or carbonium ion polymerisation process is now clear in fundamentals if not in varied details, while Prof. H. S. Taylor in a recent Spiers Memorial Lecture saw not the reactions occurring over catalysts, but the catalysts themselves as the principal object for future work in the next few years. This implies that the present necessity is for knowledge of *why* rather than *how* the catalysts function.

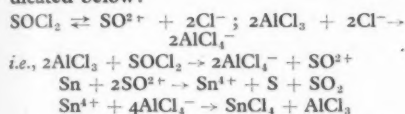
It is to be remembered that in this context a base is a substance capable of acting as a proton acceptor (Bronsted-Lowry) or electron-pair donator (Lewis), while an acid acts as a proton donator or electron-pair acceptor respectively. It is interesting to note Bjerrum's<sup>18</sup> suggestion for a rapprochement of the Bronsted-Lowry and Lewis theories—which, it will be recalled, agree on the identity of bases but not of acids—namely, that substances which act primarily as proton donators should be called acids, and those acting primarily as electron-pair acceptors or base acceptors should be termed 'antibases.'

Thus ammonia, chloride ions or electrons are bases; boron trifluoride and hydrogen ions are antibases, while com-



plexes of the type  $\text{BF}_3 \cdot \text{NH}_3$  and substances like the hydrogen halides are acids; in other words, an acid is formed from a base plus an antibase.

The view held by certain recent workers that the  $\text{AlCl}_3$ -HCl catalyst couple acted through the formation of the stable acid  $\text{HAlCl}_4$  must now be considered untenable through the careful work of Brown and Pearsall,<sup>19</sup> who could find no evidence of the existence of this compound (limit of detectability was 0.2 mol% in the mixture) even at  $-120^\circ\text{C}$ . with or without traces of water vapour or hydrocarbons. The ion  $\text{AlCl}_4^-$  does occur however. Korshak and Lebedev<sup>20</sup> suggest that the important step in aromatic reactions catalysed by halides including formation of complexes between reactants and catalyst, is the formation of the strongly dipolar dimer such as  $\text{AlCl}_2^+ \text{AlCl}_4^-$  which induces strong polarisation in the reactant molecules. In this connection it has recently been shown<sup>21</sup> that these Friedel-Crafts catalysts will catalyse the reaction of metals with thionyl chloride by increasing the concentration of thionyl ions available for reacting with the metal through reaction with the chloride ions to form the complex anions as indicated below:



As far as polymerisation processes are concerned, the evidence is now strongly weighted in favour of initiation through formation of a carbonium (or oxonium) ion from the monomer by proton donation from the acid catalyst. (With basic catalysts the monomer loses a proton to form a carbanion.) Ordinary acids act directly as proton donors, while the 'Lewis acids' ( $\text{BF}_3$ , etc.), from the work of Plesch,<sup>17</sup> Evans<sup>22</sup> and others, all appear to require a co-catalyst—which may in some cases be identified with the so-called promoters—generally a hydrogen-containing substance with which the catalyst can form a complex which can act as a proton-donating acid in the Bronsted sense. In this respect, two additional suggestions of relevance have recently been made. It has been postulated<sup>23</sup> that the homologation of alcohols with excess synthesis gas which occurs over a cobalt catalyst at  $185^\circ\text{C}$ . is due to the action of cobalt hydrocarbonyl,  $\text{HCo}(\text{CO})_4$ , which is a strong acid; some support for this has been obtained from studies on the pinacol-pinacolone reaction, where a definite catalytic action of the hydrocarbonyl itself for the transformation of pinacolone to pinacol alcohol has been demonstrated. In the other case, Parravano<sup>24</sup> has shown that polymerisation of methylmethacrylate can be initiated by the hydrogen which is present during electrolysis of aqueous solutions containing the monomer. The efficiency of the process increases with increasing hydrogen-overvoltage of the cathode metal, as indicated

Table 1. Co-catalysts proved necessary for various monomer-catalyst polymerisations (from Plesch<sup>17</sup>)

Monomer	Catalyst	Temp. $^\circ\text{C}$ .	Solvent	Proved Co-catalysts
Ethylene	$\text{AlCl}_3$	10 to 50	50% EtOH	$\text{H}_2\text{O}$ , HCl
Propene	$\text{AlBr}_3$	-78	<i>n</i> -butane	HBr, EtBr
<i>Isobutene</i>	$\text{BF}_3$	Room to 100	Gas phase none	$\text{H}_2\text{O}$ , HAc, <i>t</i> -BuOH, $\text{H}_2\text{O}$
	$\text{TiCl}_4$	-100 to 20	Hexane	$\text{H}_2\text{O}$ , $\text{H}_2\text{SO}_4$ , di- & tri-chloroacetic acid
Di- <i>isobutene</i>	$\text{SnCl}_4$	-80	Ethyl chloride	$\text{H}_2\text{O}$ , HCl
Styrene	$\text{BF}_3$	room	Gas phase	$\text{H}_2\text{O}$ , HAc
	$\text{SnCl}_4$	25	$\text{CCl}_4$	$\text{H}_2\text{O}$ ?
			Ethyl chloride	$\text{H}_2\text{O}$ , HCl
			Ethylene dichloride	Solvent?
	$\text{TiCl}_4$	-60 to 20	Hexane or toluene	Trichloroacetic acid, ethylene dichloride
		-30 to 30	Ethylene dichloride	Solvent?

by the fact that polymerisation is complete in 2, 6 and  $>24$  hr. with Pb, Zn, and Pt respectively, the corresponding hydrogen-overvoltages for which are 1.09, 0.75, and 0.07 v. It should be noted that the efficiency of the metal/hydrogen ion system to act as a proton donor might be expected to be greater the greater the hydrogen-overvoltage of the metal that is, the less is the ease of discharge of protons at the cathode. Parravano also found that metals charged with hydrogen either electrolytically or by sorption initiated polymerisation, powders or blacks being more efficient than compact metal. The overall efficiency however, was low compared with the total hydrogen released by electrolysis or sorbed.

#### The co-catalyst

Aspects relative to the co-catalyst will now be considered in greater detail, drawing largely on the results of Evans and co-workers and the excellent review of Plesch. The impetus for attention to this question, at least in the case of polymerisations, originated in the observation that a trace of an impurity in *iso*-butene or the Friedel-Crafts catalyst ( $\text{BF}_3$ ) was absolutely necessary to get any polymerisation at all at room temperature or  $-80^\circ\text{C}$ ., and that water (less than 0.1%), *t*-butyl alcohol or acetic acid were suitable impurities. It had been known for some time previously that similar impurities or 'promoters,' were beneficial in Friedel-Crafts reactions and isomerisations, alkylation, etc., involving halide catalysts. In such cases Plesch emphasises the misuse of the term promoter which implies a substance which speeds up a reaction which could still occur in its absence, whereas the co-catalyst is absolutely essential for the reaction to occur at all. He further points out that in no single instance is there yet definite proof that the halides by themselves can initiate polymerisation. Remembering the minute amounts of co-catalyst that will suffice and the difficulty of ensuring extreme purity, particularly freedom from traces of hydrogen-containing substances, it is an open question whether the co-catalyst action now recognised as essential to polymerisations with the Lewis type of acids may not be essential to all reactions involving these substances as catalysts.

The known cases of proved co-catalysts

are summarised in Table 1, for polymerisation processes. The current idea is that the catalyst and co-catalyst form an ionisable complex consisting of a hydrogen cation (proton) and a complex anion, and that the proton can be donated to a carbon (or oxygen) atom at a double bond to give a carbonium (or oxonium) cation which initiates the chain reaction. The actual nature of the co-catalyst may materially influence the rate of chain initiation and the rate and mode of chain termination; similar influences on rates and on relative occurrence of side reactions may result in reactions other than polymerisations.

In many cases the complexes have been prepared. Thus Evans refers to  $\text{BF}_3 \cdot (\text{CH}_3\text{COOH})_2$  a crystalline solid over which *iso*-butene can be polymerised and to the solid boron trifluoride dihydrate which exists as  $(\text{H}_3\text{O})^+(\text{BF}_3\text{OH})^-$ . He has also shown from absorption spectroscopy measurements that the carbonium ion  $(\text{C}_6\text{H}_5)_2\text{C}^+\text{CH}_3$ , with characteristic absorption peak at  $431 \mu$ , occurs on treating diphenyl ethylene either with sulphuric acid or moist  $\text{BF}_3$  in benzene, being formed in both cases by a proton addition to the  $(\text{C}_6\text{H}_5)_2\text{CH}=\text{CH}_2$ .

Plesch distinguishes between three broad groups of co-catalysts.

(1) Those which form strong acid complexes with the catalyst of the general type (hydrogen ion)<sup>+</sup>(catalyst-acidic part of co-catalyst)<sup>-</sup>, for example,  $\text{H}^+(\text{TiCl}_4 \cdot \text{O}_2\text{CCl}_3)^-$ ,  $\text{H}^+(\text{SiF}_6)^-$ , and aluminosilicates of use as cracking catalysts.

(2) Alkyl halides of the type R-X which, with the metallic halides of type  $\text{MX}_n$ , give the hypothetical acids of type  $\text{HMX}_{n+1}$  which provide free alkyl ions  $\text{R}^+$  and these initiate polymerisation. This is suggested as the mechanism in halogenated solvents, and is supported by the fact that styrene is not polymerised by  $\text{BF}_3$  in methyl chloride with which, for steric reasons,  $\text{BF}_3$  cannot form a complex.

(3) Hydrogen halides, which can be active, inactive or inhibitive depending on the particular system. There is no evidence for the existence of complex acids of the type  $\text{HMX}_n$ , and it is therefore considered that the catalyst or the co-catalyst individually forms a complex with the monomer, then this complex reacts with



the third substance giving the chain-initiating carbonium ion.

Broadly speaking, catalyst and co-catalyst complexes appear to be formed with halides of B, Ti and Sn acting on alkenes, while catalyst-monomer complexes occur with Al halides acting on olefins in general, and with halides of B, Ti, Sn and others acting on arylenes; then the olefin-halide complex, for example, reacts with hydrogen halide to form a monomer carbonium ion and a complex anion. In support, there is evidence that while Al halides can form complexes with double bonds, halides of B, Ti and Sn cannot form complexes either with double bonds or with the hydrogen halides—which cannot therefore be expected to act as co-catalysts in these cases.

### Esterification of olefins

At first sight, these last views of Plesch regarding polymerisation appear to conflict with some interesting results of Morin and Bearse<sup>25</sup> on the improved results in the esterification of olefins with organic acids, where a mixed boron trifluoride-hydrogen fluoride catalyst proved better than either component of the mixture by itself. They state that while this process is many years old and very attractive (because of the possibility of utilising olefins from refinery by-products, and because it is more rapid and allows easier recovery and purification of esters and catalysts than the commercially favoured esterification of alcohols with sulphuric acid catalyst), it had not been applied commercially. This was because the available catalysts ( $H_2SO_4$ ,  $BF_3$ , both pure and as dihydrate,  $TiCl_4$ , etc.) either gave low yields, required long reaction times, needed high temperatures or had to be used in large amounts.

A potentially efficient and economic process has been devised using as catalyst a 3% solution of ice-cold boron trifluoride in ice-cold glacial acetic acid to which an equal weight of anhydrous hydrogen fluoride was afterwards added, but before introduction of the olefin (otherwise free  $BF_3$  tended to polymerise the olefin and little esterification occurred). The olefin was introduced under pressure into the catalyst solution contained in a bomb of 316 stainless steel (most resistant to fluoride attack) maintained at about 50°C. for higher olefins, 100°C. for propylene and 150°C. for ethylene—which also required 10% catalyst concentration. The exothermic reaction required 15 to 30 mins. for completion. Best yields were obtained with propylene—80, 70 and 84% ester respectively with acetic, formic and chloroacetic acids. With acetic acid the yields with ethylene, propylene and butylene were 50, 80 and 50% respectively. With less than 1 wt.% catalyst there was no reaction; equal weights of  $BF_3$  and HF proved better than equal molecular concentrations, and 3 wt.% of each was the lower practical limit. It was essential to introduce the  $BF_3$  as a pre-formed com-

Table 2. Per cent. isopropyl acetate yield from propylene and acetic acid with different catalysts (Morin and Bearse<sup>25</sup>)

Catalyst	Concn. wt. %	Temp. °C.	Time, hr.	% Age Yield
$BF_3$	3	100	0.5	0
	3	150	6	37
HF	3	100	7	20
	13	100	1	55
$BF_3/HF$	3 each	100	0.5	80
$BF_3/HCl$	3 each	100	0.5	64
$BF_3/H_2SO_4$	3 each	150	5	60
Thioboric acid	3	100	1.5	40
$AlCl_3/HCl$ , $H_2SO_4$ , $ZnCl_2$ and others, under similar conditions ...				0 to 5

plex with the polar organic acid, otherwise the free trifluoride polymerised the olefin. It was further found that water considerably reduced the ester yield—from 80 to 56% with 1% water. This fact emphasises the difference in behaviour here from that in polymerisation processes indicated by Plesch. However, there is the strong synergistic effect of the dual combination as is readily seen from Table 2. It is still possible that this effect may conform to the general pattern—one of the two fluorides forming complexes with one of the two reactants—especially as the catalysts appear to be recovered as compounds of the type  $(R.CO_2H)_2.BF_3$  and *iso*-propyl fluoride.

### Polymerisations

Hamann<sup>26</sup> has reviewed at length the general types of polymerisation processes occurring through ionic initiation. The data for Table 3 is taken from his paper. He has also collected together available data on the effect of catalysts on the course of co-polymerisation processes, which emphasise the difference in the catalysis by peroxides initiating free radicals and that by acids initiating cations. The relative degree of cross linking of the two monomers is much greater with the free radical mechanism than with the carbonium ion mechanism, where the resulting polymer tends to contain a proportion of one monomer unit much greater than in the original mix. Thus, with chloroprene-styrene co-

polymers formed from a mix containing 80 mol % chloroprene, the radical and cationic mechanisms give polymers with approx. 90 and 25% chloroprene respectively.

The oxidic type of catalysts have also received much attention in the last year. They have been investigated in what is claimed<sup>27</sup> to be the first full study of the production of aromatic amines by the catalytic vapour-phase reaction of alcohols and amines—a process which permits continuous operation. A study was made of the relative efficiency of a large number of catalysts for the preparation of dimethylamine at 360°C.—alumina in lump, powder, pelleted and supported forms, phosphoric acid suitably supported, kaolin silica gel, titania zinc oxide, and chromium sesquioxide were among those tried. Alumina, titania and phosphoric acid, were the best, but all gave some nuclear alkylation, which was least with alumina. Up to 90% conversion of primary to tertiary amine was obtained, but in no case was the product pure enough, and it was concluded that more highly selective catalysts would be necessary.

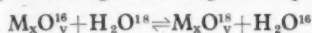
### Cracking catalysts

Two useful reviews of cracking catalysts have appeared. The first<sup>28</sup> is a reprint of a lecture by Prof. Garner of Bristol, in which he surveyed recent work from his and other schools seeking for the common links between catalysis and other reactions of solid oxides. The emphasis is on the

Table 3. Possibility of radical and ionic polymerisation of various monomers (from Hamann<sup>26</sup>)  
(1 = strong, 2 = moderate, dash = little possibility)

Monomer	Radical	Cationic Poss.	(Carbonium ion) Catalysts	Anionic Poss.	(Carbanion) Catalysts
Ethylene	1	2	Halides with co-cats.	—	
Butadiene	1	—		2	Alk. metals
<i>Iso</i> -butylene	—	1	<i>Ibid</i>	—	
Styrene	1	1	$AlCl_3/EtCl$	2	$NaNH_2/liq. NH_3$
Vinyl ether	?	1	Halides, common acids, silicates	—	
Vinyl ester	1	—		2	
Vinyl halides	1	—		—	
Acrylic esters	1	—		1	Grignard reagents in ether
Methacrylates	1	—		1	
Acrylonitriles	1	—		1	
Methacrylonitriles	1	—		1	<i>Ibid</i>
Nitro-olefins	—	—		?	
Ethylene oxide	—	1	RX-oxonium ion	—	
Ethylene imine	—	1	RX-nitronium ion	—	
Tetrahydrofuran	1	2	RX-oxonium ion RX = $HClO_4$ , etc., $FeCl_3$ , etc./co-catalyst	—	
Cyanosorbic acid	—	—		1	Alk. metals

picture of a solid oxide as an ionic lattice containing a number of defects which may arise simply through voids of metal or oxygen ions in a single substance, or through incorporation in it of other metal ions existing in a different valency state such as occurs with alumina-silica or zinc oxide-chromium oxide systems. The result is that a surface condition is attained which facilitates electron transfer processes. Haensel and Sterba<sup>28</sup> conclude that there is now general agreement on the carbonium ion mechanism of catalytic cracking but none on the actual way in which the necessary acidity of the catalyst is attained. They summarise four interesting papers on isotopic exchanges over oxidic cracking catalysts. Mills and Hinden found that oxygen exchange according to the pattern



was rapid and reasonably large with silica gel, alumina gel and silica-alumina catalysts, but there was negligible exchange with un-activated kaolin and bentonite clays which, however, allowed exchange after activation with sulphuric acid; the conclusion was that the activated material has a higher surface area and a more labile surface which permits the change in co-ordination number of the aluminium from 6 to 4, as was postulated for a good cracking catalyst. From studies on the hydrogen-deuterium exchange and the hydrogen transfer from decalins to butylenes over silica-alumina mixtures of varying composition, Blue and co-workers have concluded that the transfer occurs largely through an ionic mechanism over an acid catalyst rather than as a result of adsorbed hydrogen atoms.

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## Advances in Catalysis

THE present volume\* is a worthy successor to the first two volumes of this series. Once again the editors have covered a wide part of the field of investigation into catalysis, ranging from fundamental studies to specific technical applications.

The first article, 'Balandin's Contribution to Heterogeneous Catalysis,' by B. M. W. Trapnell, while emphasising the importance of Balandin's contribution to the theory of catalytic action on surfaces, at the same time demonstrates the inadequacy of the geometrical approach to the explanation of the phenomena. The article would have been more appropriate in one of the earlier volumes, as Beek's article on 'Catalysis and the Absorption of Hydrogen by Metal Catalysts' in Volume II gives a more up-to-date and complete picture.

'The Poisoning of Metallic Catalysts,' by E. B. Maxted, is a review of the work on the effect of sulphur compounds on the

hydrogenating efficiency of platinum and nickel catalysts, which has been going on at Bristol University for many years. The technical man will recognise some of Maxted's methods of detoxication as the basis of the widely applied 'guard tube' method for the protection of expensive catalysts.

P. W. Selwood's article, 'Magnetism and the Structure of Catalytically Active Solids,' is an excellent survey of recent developments in the study of the structure of supported metal catalysts by magnetic methods. The method is elegant, but, given the magnetic equipment, the technique is not so exacting as to exclude magnetic methods from the examination and the control of the manufacture and application of catalysts on a technical scale. In fact, the technique is so powerful that one would expect a rapid expansion of its application for these purposes.

'Catalytic Cracking of Pure Hydrocarbons,' by V. Haensel, and 'Chemical Characteristics and Structure of Cracking Catalysts,' by Oblad, Milliken and Mills, are complementary articles and will repay

the attention of non-specialists, as well as of those whose interests lie in hydrocarbons. They cover the developments of the past 15 years in the cracking of hydrocarbons, leading up to the present state of progress where 'cat-crackers' in which fluidised catalyst is circulated and regenerated at rates of many tons a minute are a feature of every major oil refinery. The mechanism of hydrocarbon reactions on cracking catalyst is now well understood as a result of the efforts of organic and physical chemists and physicists working in collaboration. Cracking catalysts are recognised as 'structural acids' and, once again, carbonium ion theory is adequate to explain the hydrocarbon reactions which occur.

The article by Wheeler on 'Reaction Rates and Selectivity in Catalyst Pores' forms a link between the articles on cracking, on acid catalysts, and catalysis by metals. It was early recognised that catalysts must be porous if their large surface area is to be accounted for. This article gives the mathematical treatment of the subject and reviews the experimental results which are in close accord with expectation, and gives a rational explanation of catalyst selectivity and poisoning.

The 'Nickel Sulphide Catalysts,' by Kirkpatrick, gives a comprehensive survey of the many uses to which these versatile catalysts may be applied: hydrogenation, dehydrogenation, isomerisation, disproportionation, etc.

The article 'Catalytic Oxidation of Acetylene in Air for Oxygen Manufacture,' by Rushton, deals with a particular technical problem. It is, however, of general interest in indicating how the increasingly large scale of application of a known process can lead to a new and serious problem (accumulation of traces of acetylene from the air) and the fantastically high space velocities at which catalysts may at times be called upon to operate.

'Advances in Catalysis' may be commended both to the specialist and to the person who will read the book because catalysis is a fascinating subject.

P. J. GARNER, B.A., PH.D., F.INST.PET.

**Engineering a petroleum-chemicals plant.** Three technical papers, 'Some Engineering Aspects of Building a Plant for the Production of Chemicals from Petroleum,' 'Fuel, Heat and Power Aspects of the Petroleum-Chemicals Plant for Petrochemicals Ltd.,' and 'Chemical Engineering Aspects as Applied to the Building of a Petroleum-Chemicals Plant,' read, respectively, before the Manchester Association of Engineers, the Institute of Fuel and the Institution of Chemical Engineers, have aroused so much interest that they have now been brought together in the convenient form of an illustrated brochure published by Petrocarbon Ltd. The whole constitutes a comprehensive description of the new works at Partington.

\**Advances in Catalysis*. Edited by Frankenburg, Komarewsky and Rideal. Vol. 3. Academic Press, 1951, pp. 360 inc. index, \$7.80.

# A Modified 100 cc. Othmer Equilibrium Still

By S. R. M. Ellis, Ph.D., A.M.I.Chem.E.

(Lecturer in Chemical Engineering, University of Birmingham)

A large number of papers has been published on the relative merits and disadvantages of a great variety of equilibrium stills of the differential, batch recycling and continuous types. The majority of these stills require a bulk sample of 250 to 500 c.c. of mixture for an individual equilibrium determination, which means that for the complete determination of an equilibrium curve large amounts of the pure components are required. In practice this is often a very serious difficulty and in this short paper\* an equilibrium still requiring a charge of only 100 cc. is described.

THE still described is a modification of an Othmer still designed by K. F. Coles, of Trinidad Leaseholds Ltd., to take a charge of 100 cc. The advantage of Coles' equilibrium still was the use of smaller quantities of liquid mixture and increased accuracy because of the small condensate fraction (0.6% of the original charge). In experiments with this still on several binary mixtures it was observed that the returning condensate tended to stream through the liquid in the still and concentrate in the surface layers. The heater tube recycled mainly the liquid at the bottom of the still. In other words, the turbulence was insufficient and incorrectly directed to give efficient mixing of the still contents.

A new type of circulating device has been devised and incorporated in the modified still described in this paper. This still is illustrated in Fig. 1, and the essential general details are as follows:

A Glass shell containing the boiling liquid mixture.

B Insulated boiling tube with a 60-ohm 32-gauge Brightray winding for external heating.

C Two 5-mm. i.d. inlet downtake holes such that the liquid in the still moves into these and then down the central 6-mm. i.d. tube D to the bottom of the boiling tube B. There the liquid mixes with the incoming condensate in the 2-mm. capillary return line, and moves up to tube B to E, is boiled, and then overflows in a fountain on to the surface of the liquid in the still. This circulating device gives even boiling and has been very satisfactory. If desired an internal heater can be used.

F Thermometer pocket for the measurement of liquid boiling point temperatures.

G Externally heated column similar to that used in the standard Othmer still. The heater was a 150-ohm 32-gauge Brightray resistance wire.

H Condenser.

I Receiver of approximately 0.6 cc. capacity.

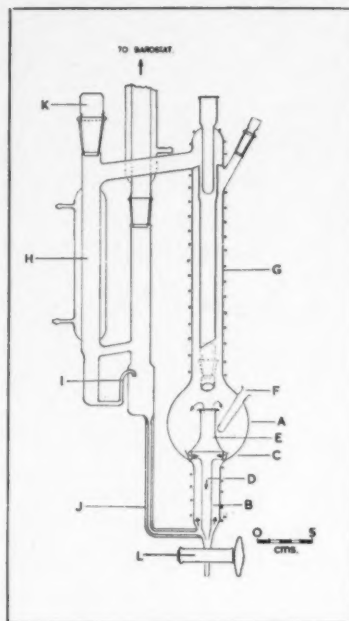


Fig. 1. Modified still.

J Capillary return line (2 mm.) to the bottom of boiling tube B.

On shutting down the still, stopper K was removed and the liquid condensate

sample obtained by means of a pipette or hypodermic needle. The still liquid was similarly sampled from a removable stopper on the still.

## Experimental results and discussion

The following experimental results have been determined for the system ethyl alcohol-benzene (see Table 1).

The experimental log activity-coefficient data have then been plotted against composition as shown on Fig. 2. From the experimental log activity-coefficients at infinite dilution theoretical activity-coefficients have been calculated using the Margules 3-suffix equation.

The activity-coefficients from Table 2 have also been plotted on Fig. 2.

A comparison of the two plots shows there is close agreement between the experimental and correlated results. Thus for such a system the experimental results are likely to be accurate.

When, however, the still was used for determining the equilibrium data on systems such as ethyl alcohol-nitrobenzene or benzene-nitrobenzene there was a discrepancy between the experimental and theoretical activity-coefficient plots. This was attributed to the fact that, in having to shut the still down to take samples, some degree of fractionation of the vapour occurred.

Table 1. Ethyl alcohol-benzene

Mol. % Ethyl alcohol			Ethyl alcohol			Benzene		
t°C	x	y	P <sup>E</sup>	Z	log Zγ (alcohol)	P <sup>B</sup>	Z	log Zγ (benzene)
70.0	14.0	34.0	0.7106	0.991	0.5264	0.7263	0.986	0.018
69.0	20.0	37.6	0.6776	0.990	0.4388	0.7026	0.985	0.039
68.0	32.8	42.4	0.6500	0.989	0.2938	0.6789	0.984	0.094
68.0	60.6	50.6	0.6500	0.989	0.1022	0.6789	0.984	0.259
69.0	72.0	55.8	0.6776	0.990	0.054	0.7026	0.985	0.345
70.0	79.0	60.6	0.7106	0.991	0.029	0.7263	0.986	0.406
72.0	87.9	70.5	0.7738	0.994	—	0.7738	0.989	0.494

Table 2

Mol. % Ethyl alcohol x	A	B	Ethyl alcohol log γ (alcohol)	Benzene log γ (benzene)
0.20	0.73	0.63	0.49	0.03
0.30	0.73	0.63	0.32	0.07
0.50	0.73	0.63	0.155	0.18
0.60	0.73	0.63	0.095	0.255
0.80	0.73	0.63	0.02	0.425

\* Reproduced, by permission, from *Birmingham University Chemical Engineer*, 1951, 3 (1), pp. 34-36.



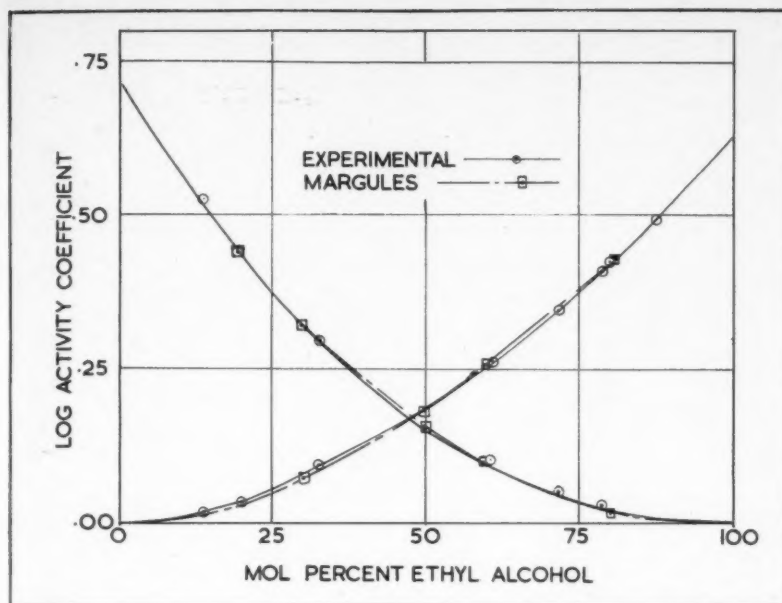


Fig. 2. Experimental and correlated activity-coefficients.

This is more marked with systems with a large difference in boiling points between the components. To overcome this difficulty further modifications have been made by enlarging the condensate receiver and having sample taps on the condensate receiver and on the still A. Thus, while the still is operating, samples of the vapour condensate and the liquid in the still can be taken simultaneously.

#### Conclusion

For binary mixtures with a small difference in boiling point between the components the modified 100-cc. equilibrium still described in this article gives accurate equilibrium data. Such a still requires relatively small amounts of liquid, is easily handled, and enables equilibrium to be rapidly attained.

## Dyes Improve Solar Evaporation of Salt

In India it has been found that the solar evaporation of sodium and magnesium chloride solutions may be accelerated by the addition to the solutions of traces of infra-red-sensitive dyes. The results of these investigations and their significance in the commercial production of industrial-grade salt under otherwise unfavourable conditions are discussed below by G. P. Kane and G. R. Kulkarni, Chemical Technology Department, Bombay University.\*

TWO important requirements for the successful operation of a solar salt industry are the availability of brine of adequate concentration and a warm dry season of considerable length. On the West Coast of India almost the whole of the annual rainfall occurs in the four months from June to September, after which a fairly long dry season is available for the solar evaporation of sea water in ponds and for subsequent working up of the deposited salt. On the East Coast, however, the rainy season extends to January, and the salt content of the sea water is lower because of constant dilution by fresh water from many rivers. The favourable conditions of the West Coast are responsible for the large amount of salt produced there by solar evaporation, while in Bengal, on the East Coast, the production of salt by this method is almost negligible. If, however, the available dry season could be made more effective for salt production by achieving a greater rate of solar evaporation by some means or other, this method of producing salt might also be used on the East Coast. In recent years attempts have been made to achieve this by adding dyes to the salt solutions.

The rate of evaporation is proportional to the absorption of solar radiation falling on the exposed surface of the liquid.

\* Summarised from the 'Transactions of the Indian Institute of Chemical Engineers,' 1949-50, III, pp. 104-108.

Usually a large proportion of the non-penetrating infra-red heat radiation is reflected away by the colourless liquid which acts as a mirror. Increased absorption and, therefore, evaporation can be obtained by adding green or red dyes which will absorb such radiation more effectively.

#### Infra-red-sensitive dyes

High solubility, good infra-red absorption properties, light fastness, non-toxicity and low cost are some of the characteristics which determine the suitability of a dye for accelerating the rate of solar evaporation. One of the two dyes selected was a basic dye, Astraphloxine FF, belonging to the cyanine group and used extensively in infra-red photography. Astraphloxine FF gives a deep pink solution even when only 1 part in 100,000 is added to the solution. The other was an acid dye, Naphthol Green B, which gives a deep green solution. The light fastness of Astraphloxine FF and Naphthol Green B is 2 and 7 respectively, on a scale where an excellent light fastness is represented by 8. Commercial samples of the dyes were used by the authors for these experiments.

#### Tests with salt solutions

Solutions containing about 3% sodium chloride and 20% magnesium chloride, respectively, were employed for the experiments. For each experiment, 200 c.c. salt solution were placed in a shallow enamelled

rectangular dish of identical size. The requisite amount of dye was added to one dish and both were then exposed to the sun on the roof between 11 a.m. and 3 p.m. during the winter months, November to February. During this period there is a steady north-westerly breeze in Bombay and the variations in humidity are not great.

The loss of water by evaporation was determined every hour and also for the total period of exposure by weighing the dishes as well as by titration of samples of the solution with N/10 silver nitrate. To determine the effectiveness of the dye over prolonged periods the dishes were kept covered overnight and the solution was made up to 200 c.c. with distilled water. The evaporation was repeated in the same manner on successive days. The dyes were added in the proportion of 5, 25 and 100 g., respectively, to 100,000 c.c. of the solution to determine the influence of concentration.

The results of the experiments with sodium chloride solutions show that initially both Astraphloxine FF and Naphthol Green B accelerated the rate of evaporation appreciably, even though the former was used in much lower concentrations. However, the colour of the Astraphloxine began to fade very soon and its effect declined to zero. With Naphthol Green B, the rate of evaporation increased at first by as much as 30% and even on the

sixth day its effect was appreciable. The low cost of the latter and its effectiveness over a fairly long period indicate that it may prove satisfactory for use in solar evaporation on a large scale.

Naphthol Green B alone was employed in the experiments with magnesium chloride solutions. In this case it was observed that when the dye was added in the proportions of 25 to 100 g. to 100,000 c.c., the solution became turbid, probably because of the precipitation of the corresponding magnesium salt. The results obtained were therefore somewhat erratic. With only 5 g. of the dye per 100,000 c.c., however, the solution remained clear and the percentage increase in evaporation gradually decreased as in the case of the sodium chloride solution.

These preliminary experiments have demonstrated that the solar evaporation of sodium or magnesium chloride solutions may be accelerated between 10 and 20% by additions of very small amounts of Naphthol Green B. However, since neither of these dyes is edible, the process can only be used for the production of industrial salt.

These observations are in general agreement with an I.C.I. report that in Australia increases in the yield of salt up to 20% could be expected from existing crystallisers by colouring the final evaporation ponds with *Solivap Green*.

### Oil tank cleaning

The Groom system of oil tank cleaning is claimed to be the answer to the hitherto long and costly process of tank cleaning. Until now two methods have been generally used: manual labour and various systems of washing out the tank with boiling water. In both systems none of the oil is reclaimed, the methods are expensive and, in the latter case, the equipment is heavy and unwieldy.

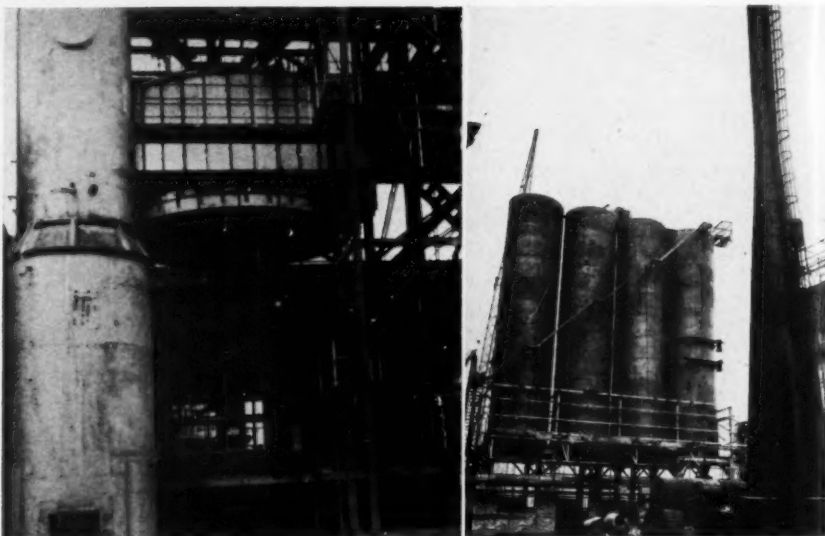
In the new system developed by the Groom Patents Co. an aqueous detergent solution is heated and pumped to the point of use, where it is discharged from either a manually operated or a specially automatic projector against the surface to be cleaned. The hot detergent acts upon the deposit and removes this by a combination of dissolution, reduction of interfacial tension and scouring.

The mixture of oil, deposit and used detergent flows to the lowest point of the tank and is pumped from there to a separating and heating tank. (The latter is situated at a central point, for instance, on deck if it is permanently fixed, or on a barge or lorry.) In its course through this tank the detergent solution separates from the oil and deposit, is heated and again pumped to the point of use, thus completing the cycle. Any loss of detergent solution (theoretically nil) is automatically made up. Coincidentally, cleaned oil is reclaimed and automatically expelled from the tank.

## New Oil Catalyst Plant built by Crosfields at Warrington

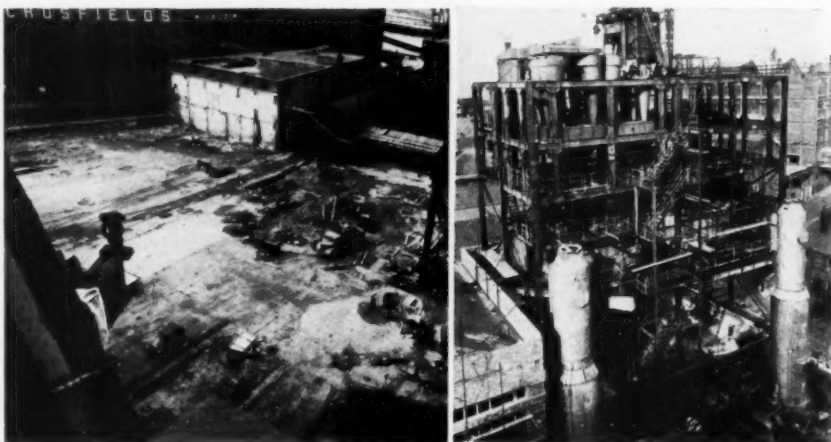
A £1,000,000 plant for the manufacture of catalysts for the cracking of petroleum has been built at Warrington, Lancashire, by Joseph Crosfield and Sons Ltd., a Unilever company, in co-operation with the Davison Chemical Corporation, Baltimore, U.S.A., and as a result of agreements with five big oil companies: Esso Petroleum Co. Ltd., Anglo-Iranian Oil Co. Ltd., Shell Refining and Marketing Co. Ltd., Trinidad Leaseholds Ltd., and Bahrein Petroleum Co. Ltd. The plant operates by the process developed by the Davison Chemical Corporation. A large part of the design and construction work was carried out by E. B. Badger & Sons (Great Britain) Ltd., and Taylor Woodrow Construction Ltd. Construction was started in March, 1950, and the plant came into operation at the end of 1951.

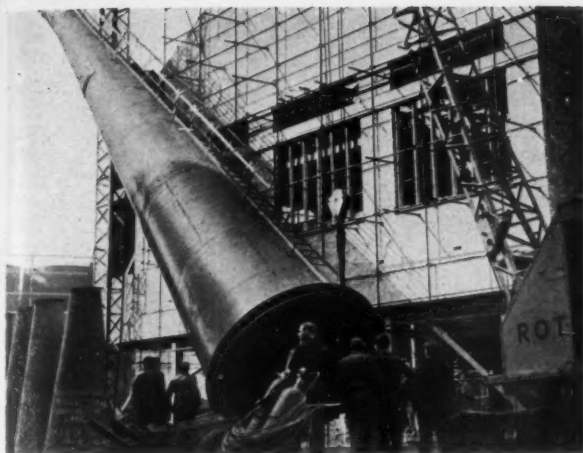
The main raw material is sodium silicate. Spray drying is probably the most important part of the process since it provides a catalyst of more uniform particle size than was obtainable by earlier methods. This size may be controlled to suit the requirements of any particular oil company. The equipment includes two Swenson Evaporator Co. stainless steel driers, which have been fabricated under licence in the U.K. They are the largest type of these driers which have been made; each vessel is 22 ft. I.D., 44 ft. high and 45 tons in slung weight. The catalysts produced in this new plant will be used in the "cat crackers" of the new oil refineries built or being built in various parts of the U.K. in order to make the country self-sufficient in oil-refining capacity. This self-sufficiency programme will save dollars and the new catalyst plant will add to the savings.



Above: (Left) The upper part of one of the spray driers, with the wind box suspended and the upper part of the cone below. The hot gases which are produced in the Peabody furnace pass into the wind box inlet and are distributed round the body of the drier through slots in the side of the chamber. (Right) The finished product silos and secondary drier stack

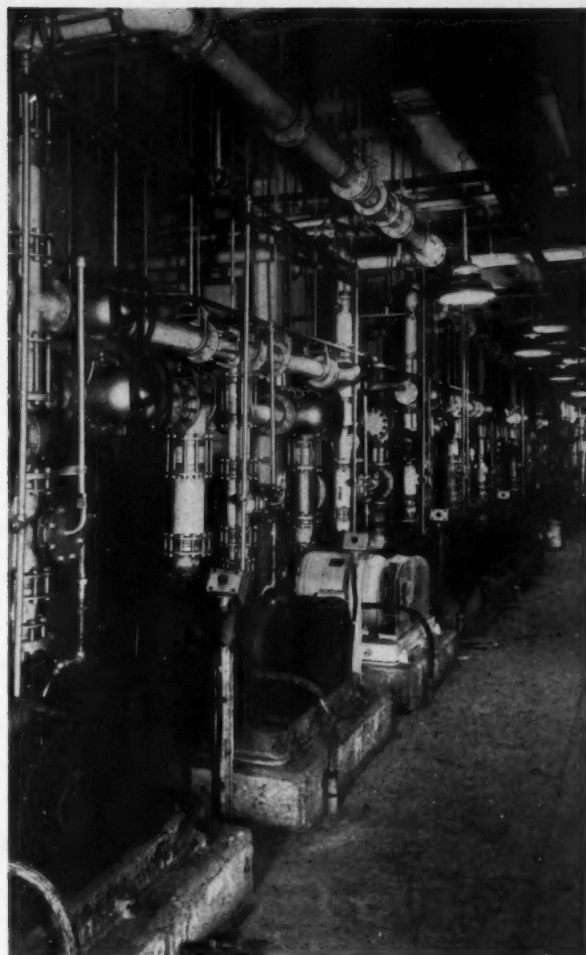
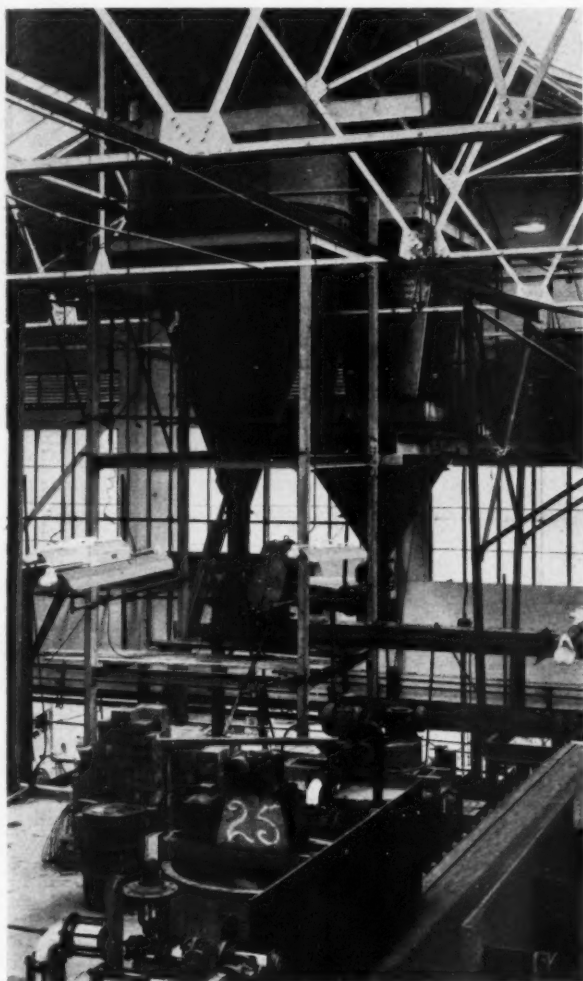
Below: On left, the cleared site at an early stage of construction in 1950 with piling in progress. On right, the erection of the spray driers showing structural steelwork.





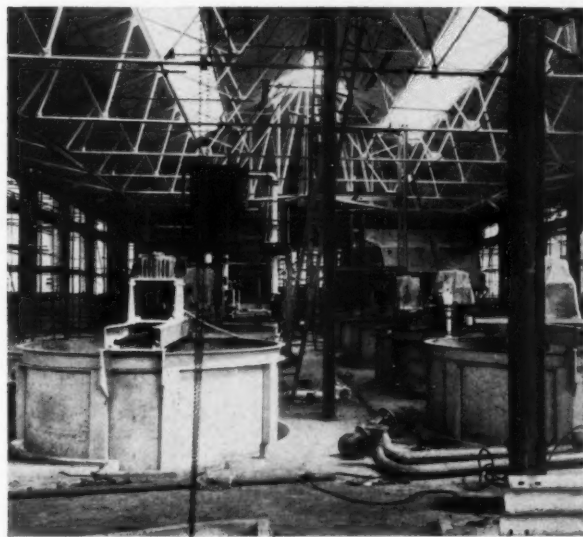
**Above :** Erection of the final drier stack in September of last year. The complete plant came into operation at the end of last year and is expected to lead to the saving of several million dollars.

**Below :** Erection of the secondary drier equipment in October, 1951. Design work for the plant was begun in March, 1950. With a few exceptions, it has been constructed and equipped with materials of British manufacture.



**Above :** The line of slurry transfer pumps and pipelines. The silica-aluminium slurry is pumped by high-pressure plunger pumps through a series of nozzles located in the upper part of the spray drier chamber.

**Below :** A view of the first floor of the Warrington plant during construction.





# Training the Chemical Technologist

A CONFERENCE on the training of the chemical technologist was held at Bradford Technical College on February 16. It was organised by Bradford Chemical Society, with the co-operation of the West Riding Section of the Society of Dyers and Colorists and Leeds Metallurgical Society. The conference, which dealt with the training of the dyer, textile chemist, metallurgist and chemical engineer, was held in two sessions. In the first session the views of the chemical industry were put forward, while in the second the views of teachers were advanced.

The chair was taken by MR. H. RICHARDSON, Principal of Bradford Technical College. For the purpose of the conference, Mr. Richardson defined a chemical technologist as one who had had training approximately equivalent to that of a pure chemist taking a degree or equivalent qualification. He said that there could be no doubt of the importance of chemical technology, in view of the wide interest shown by professional associations, the Anglo-American Productivity Council and by the recent Government White Paper. The functions of the chemical technologist were particularly important today and the proper training of the technologist could mean much to the chemical industry.

## The dyer

MR. G. B. ANGUS, a director of John Crossley & Co., speaking on the training of the dyer from the industrial point of view, said that a well-trained dyer should have an adequate technical knowledge and the ability to use and impart it. He thought that, while a knowledge of management was important, the subject did not lend itself to teaching, depending primarily on personality. A basic knowledge of machinery and its maintenance should be taught and more emphasis placed on practical work. He complained that many technologists had an inadequate knowledge of English, both written and oral, which might lead to the misinterpretation of instructions and reports. The managerial dyer should have tact and a fair share of the important sixth sense—commonsense.

MR. W. L. THOMAS, chief chemist, Woolcombers Ltd., speaking on the training of the textile chemist, emphasised the need for chemists at all levels of training in the textile industry. It was essential to combine practical works experience with technical training. Craftsmanship and the ability to work with craftsmen should be developed. It was desirable that some general knowledge of affairs outside the laboratory, such as costing, market reports and the work of other departments, should be included in training. It was essential that the chemical technologist should be trained to express himself intelligently and concisely, both verbally and in writing.

MR. G. W. GREEN, President of Leeds Metallurgical Society, speaking on the training of the metallurgist, suggested the inclusion of industrial administration and work in industry in the normal training of the metallurgist.

## The chemical engineer

MR. TREFOR DAVIES, a director of Bantam Products, considered that the training of the chemical engineer should include university degree chemistry, physics and mathematics, together with training in engineering, plant layout, design and operation. It was important that training should be in the university atmosphere where close contact with men training for other professions could develop a broad outlook. Vacation work in chemical plants during student years was as important as work on small-scale plants in college.

In the discussion following the main speakers in the first session MR. BIRD, lecturer in dyeing, Leeds University, thought that on entering industry the young chemist should spend some time in the actual works. DR. HAPPEY, Bradford Technical College, suggested that, as in the rayon and explosives industries, young chemists should start as shift chemists and rise from this to higher positions.

## Teachers' views

Opening the second session, MR. A. W. DOYLE, Ministry of Education, pointed out that training was primarily the function of industry, education being that of the universities and technical colleges. He laid stress on the need for full-time teachers to keep themselves fully in touch with scientific and technological developments. The Ministry of Education encouraged research and the release of teachers to industry for short periods.

DR. R. L. ELLIOTT, head of the Department of Chemistry and Dyeing, Bradford Technical College, speaking on the training of the dyer and textile chemist from the teacher's point of view, said that for the adequate absorption of high polymer science into the dyeing and textile industries both primary and secondary technologists were required. The primary technologist required education in chemistry, physics and mathematics to a high standard, followed by more advanced study in one branch allied to industry, including methods of development and both theory and practice. He thought that an exclusively university training did not develop a mental outlook and tempo compatible with those of industrial production. Works visits and vacation courses in industry would help to acclimatise the student to the industrial atmosphere.

The secondary technologist required preliminary training in chemistry, physics and mathematics to intermediate degree

level, followed by a four-years' part-time course. Early presentation of technical processes and principles was necessary. Teachers must have good industrial experience and be progressive in outlook. Industry alone could enable this to be attained. Greater freedom of access of teachers to industry and a greater tendency of industry to regard the teacher as an educationalist and not merely as the operator of a production line would speed the economic development of industry.

## Engineering courses

MR. K. L. BUTCHER, senior lecturer in chemical engineering, Bradford Technical College, said that the most able aspirants to chemical engineering should follow a course similar in design to those conducted by universities and colleges for the training of engineers. Less outstanding aspirants should follow the route of the national certificates to graduate and associate membership of the Institute of Chemical Engineers. Special one- or two-year courses were available to chemists or engineers wishing to specialise in chemical engineering.

DR. N. K. PETCH, lecturer in metallurgy, Leeds University, considered that two types of technologist were required by industry. On the one hand, there was the research worker or investigator of scientific problems. For this type a degree in pure or applied science, followed by some post-graduate technological study, was required. On the other hand, there was the managerial type of technologist. The training of this type had been much discussed recently and it had been suggested that a 'liberal' education was necessary, usually meaning the inclusion of cultural subjects in the course of training. He thought it was better to rely on contacts within the university or college with, perhaps, guided reading in cultural subjects. He felt, however, that in addition to the more scientific and technological training there was a strong case for the inclusion of engineering, economics and industrial relations.

## Discussion

In the discussion following the main speakers in the second session considerable comment was made on MR. DOYLE'S suggestion that teachers be released to industry for short periods. MR. ANGUS thought that little would be gained by a teacher spending six months in a textile mill. MR. SEARLE (Bentham) suggested that the teacher might go to a research association rather than into industry. DR. ELLIOTT said that the atmosphere in a research association was totally different from that of industry. Other speakers stressed the need for industrial practice and administration in the training of the chemical technologist.

# Carriage of Dangerous Chemicals in Ships

Recommendations for the packing, storage and labelling of more than 600 dangerous substances, and for the carriage of explosives in ships are made by a committee which was appointed by the Minister of Transport to consider the existing rules. The committee, which was headed by Dr. H. E. Watts, H.M. Chief Inspector of Explosives, included a number of other chemists. No radical changes in the existing rules are proposed, but the committee urge that regulations should be as simple as possible, that they should be completely codified with the object of getting international agreement, and that a standing committee should be formed to deal with new problems as they arise and make recommendations. The report\* is being considered by the Minister of Transport.

THE rules for the carriage of dangerous goods and explosives in ships developed haphazardly, and none had been issued in detail before the first world war. A memorandum on the subject was in existence, but it merely referred to orders and recommendations issued by various Government departments. During the first world war, certain types of ammunition were permitted to be carried without a magazine, if specially packed and stowed. This concession, with an amended specification for magazines, became part of the Dangerous Goods Memorandum issued by the Board of Trade in 1924.

In 1930 a committee was appointed by the Board of Trade to consider the whole question, and its report, issued in 1933, was adopted with some amendments and additions. The rules were stated not to apply to Government explosives when shipped under regulations laid down by the department concerned, but were adopted with few changes by the Sea Transport Department for controlling the carriage of service explosives in merchant ships.

\* 'Carriage of Dangerous Goods and Explosives in Ships.' Stationery Office, London, 1951. 15s.

Just before 1939, the increasing quantities of commercial explosives being shipped led to difficulties in interpreting the rules, which had been satisfactory until then. After consultations, a Sub-Committee on Sea Transport of the Explosive Storage and Transport Committee was formed. A revised set of rules for the carriage of Government explosives in freight ships was then drawn up, together with a circular describing how they could be applied to commercial explosives. This circular also revised the rules for carrying dangerous goods, no comprehensive list of the changes and additions since 1933 having been published. The whole question was kept constantly under review, a succession of circulars being published, the last in October 1947. Before then, on July 30, 1946, the new committee which issued the present report had been appointed. They found their predecessors' work to be generally satisfactory, so the rules in the various circulars and the system of classification of the 1933 report were generally adopted with few amendments.

The new committee bore the international aspect in mind when framing their report with reference to the recommendations of the International Conven-

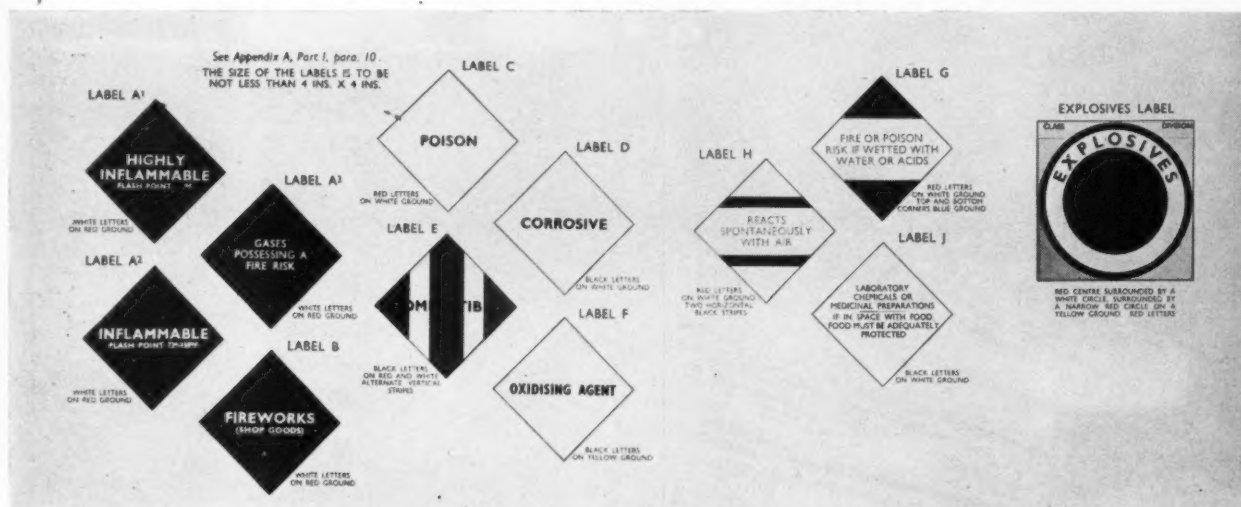
tion for the Safety of Life at Sea, 1948, and to the fact that British ships carry goods in all parts of the world. An attempt was made to draw up rules which might be suitable for international application.

British rules were examined to see whether, where they differed from the practice of any foreign country, they could be altered with advantage, and the regulations concerning the carriage of dangerous goods by merchandise train, issued by the Railway Clearing House, were also taken into consideration. The transport of such goods by land is less hazardous than by sea, so that any international system which was satisfactory for use in ships should also be applicable to inland transport. To be wholly effective it ought to be accepted everywhere.

The committee believe that no great changes in British practice are called for and that the rules they recommend could form the basis of an international code.

The system of classification adopted is substantially that used by the 1933 committee and is as follows:

- (i) Explosives.
- (ii) Compressed gases: permanent, liquefied and dissolved.
- (iii) Corrosive substances.



**LABELS FOR DANGEROUS CHEMICALS**, the sizes of which are to be not less than 4 in. x 4 in. A1, 'Highly Inflammable'; A2, 'Inflammable'; A3, 'Gases Possessing a Fire Risk'; B, 'Fireworks', all with white letters on red ground; C, 'Poison', red letters on white ground; D, 'Corrosive', black letters on white ground; E, 'Combustible', black letters on red and white alternate vertical stripes; F, 'Oxidising Agent', black letters on yellow ground; G, 'Fire or Poison Risk if Wetted with Water or Acids', red letters on white ground, top and bottom corners blue ground; H, 'Reacts Spontaneously with Air', red letters on white ground, two horizontal black stripes; J, 'Laboratory Chemicals or Medicinal Preparations—If in Space with Food, Food must be Adequately Protected', black letters on white ground; 'Explosives', red centre surrounded by a white circle, surrounded by a narrow red circle on a yellow ground, red letters.



- (iv) Poisonous substances.
- (v) Substances giving off inflammable vapours.
- (vi) Substances which become dangerous by interaction with water.
- (vii) Strong supporters of combustion and readily combustible solids.
- (viii) Substances which become dangerous by interaction with air or are liable to spontaneous combustion.
- (iv) Any other substances which experience has shown or may show to be of a dangerous character, including radioactive materials.
- (x) Medicinal and laboratory chemicals in limited quantities.

The alterations proposed generally tend to make application of the rules easier. For instance, the different degrees of risk from substances having the same type of hazard and being classified accordingly, are now given and the schedules are more detailed than formerly. Modern knowledge and experience have been taken into account and it has thus been found possible to permit the carriage of dangerous goods in passenger ships in certain cases, though important restrictions remain.

#### Gases

Stowage conditions for inert gases have been relaxed considerably as a result of experience gained in the shipment of gas cylinders since the 1933 report came out. It is realised that such substances as carbon dioxide and nitrogen do not present the same risk as, say, acetylene or carbon monoxide.

#### Corrosive substances

The recommendations for the construction of packages for corrosive substances have been so framed as to enable technical advances to be utilised as they take place, while seeking to ensure that such containers shall be perfectly safe always. Thus, if new corrosion-resisting lacquers, metal alloys and plastics continue to come on to the market, shippers will not be debarred from using them, because rigid regulations stipulate the employment of materials available at a certain date though something very much better may since have been developed.

#### Poisons and inflammable vapours

Certain poisons which do not give off toxic gases have been included because of risk of contamination of food cargoes, but a note has been added to the heading of the relevant schedule stating that there is not normally any risk associated with them.

The only major change made in the schedule of substances giving off inflammable vapours has been the addition of certain materials, including carbon disulphide, cellulose enamels and lacquers, pastes and semi-solids, which used to be dealt with in a separate list. It has not been considered practicable to give detailed packing and stowage conditions for in-

dividual inflammable substances, because of their number and the wide possible variation in the flash points of different grades of the same material.

Substances which become dangerous by interaction with air or water were grouped together in the 1933 report, but the new committee thought that the former had a closer relationship with those liable to spontaneous combustion. This particular section in the present report therefore deals only with substances likely to become dangerous by interaction with water.

#### Ammonium nitrate

Some very interesting comments are made on the recent explosions occurring in cargoes of ammonium nitrate and the very severe restrictions introduced as a result by certain authorities. The committee consider the latter to be unjustified and, following experiments carried out in this country, have come to the conclusion that commercially pure ammonium nitrate as made in Britain is not in itself an explosive and that explosions with this material have only occurred when it has been mixed with an unapproved proportion of organic material.

Dealing with potassium chlorate, the committee have not felt justified as yet in revoking the existing permission to pack the substance directly into paper-lined wooden containers or paper bags in wooden cases, since there has been only one serious accident, which occurred during the war period. It is, however, very strongly recommended that new iron or steel drums should be used for appreciable quantities if the material is not packed in glass or earthenware containers in cases.

#### Radioactive materials

Turning to radioactive materials, or substances containing it, the committee have decided that further consideration of the subject will be necessary before detailed rules can be prepared. They recommend that for the time being all proposed shipments should be submitted to the Ministry of Transport for approval.

#### Stowage and labelling

In dealing with the question of stowage, the committee have continued with their policy of simplifying and codifying the rules for the benefit of shippers and master mariners. Every effort has been made to eliminate such vague phrases as 'allowed in small quantities' and to give the exact amount permitted with precise definitions of expressions like 'away from living quarters.'

A series of labels has been designed which, it is believed, will meet the requirements of the Merchant Shipping Act, 1894, and the International Convention for the Safety of Life at Sea, 1948. The wording has been reduced to a minimum, so that the labels, like the rules generally, could form the basis of an international code. In fact, it is considered that, provided the

shape and colour of the label is always retained, the wording could be in any language and the meaning be perfectly clear anywhere in transit. The committee examined the labels agreed upon under the Berne Convention for rail transport in Europe, but found these to be too alarming. Generally they expressed the opinion that the word 'dangerous' should not be used so freely that it might come to be disregarded. Consultations were held with British Railways, but it was not found possible to agree on a common set of labels for use by rail and at sea.

Finally, it is suggested that, though the foregoing rules should be statutory, they should also be flexible. Provision should be made for shipping to be loaded in accordance with the rules prevailing at the port of destination, should these differ from British practice. Otherwise a captain, under the impression that the cargo had been packed and stowed in compliance with safety regulations, might take his ship out of a United Kingdom harbour and have great difficulty in getting it landed in the country to which it was consigned.

The committee also propose that, because a number of amendments soon becomes confusing, the entire schedules should be re-issued in amended form as often as possible, frequent changes being inevitable. They advocate the setting up of a standing committee to advise on policy regarding dangerous goods. The subject is one of increasing complexity and the present system, whereby the Home Office advises on commercial explosives, the Explosives Storage and Transport Committee on Government explosives and the Government Chemist on chemicals, is not entirely satisfactory. A permanent body could discuss problems as they arose from both technical and shipping aspects in order to reach solutions which should be satisfactory to all parties concerned.

## Correspondence

### Filter output

To the Editor

DEAR SIR,

We have seen with pleasure that you were able to include one of our filters in your review of recent developments in the field of filtration (February issue). Unfortunately, we made an error when we submitted the copy for this. The error was that the output figures given are those of the Pilot, or 20-cm. filter, and the actual ones for the Princess Ariston range from 20 to 10,000 gal./day, dependent upon the number of sheets and the material being filtered. Should you have an opportunity of making this correction at any time we would be very grateful.

JOHN C. CARLSON LTD.

Ashton-under-Lyne, Lincs.



# A Cheap Atomic Reactor

**DESIGN** studies of a low energy reactor for the U.S. Atomic Energy Commission which, it is estimated, would cost only about \$1,000,000 to build, have recently been completed by North American Aviation Inc. The chief design features are summarised below.

## Design considerations

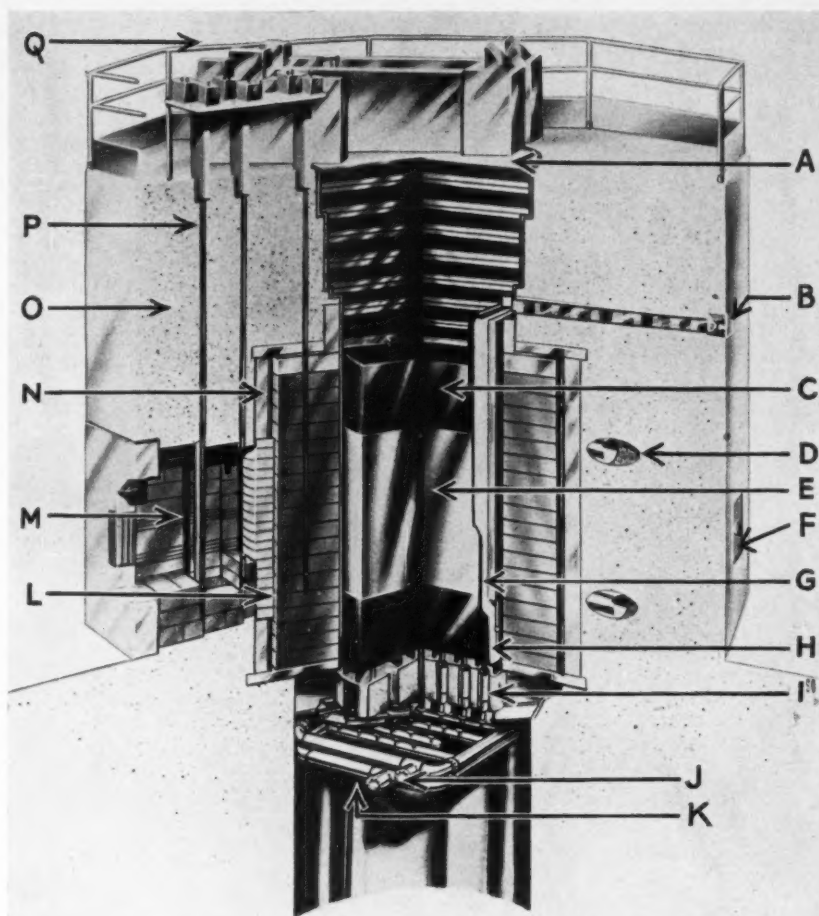
The reactor was designed by the company's atomic energy research department under Dr. Chauncey Starr for the U.S. Atomic Energy Commission's reactor development division. The reactor design study was completed originally towards the end of 1949, but engineering studies and component development work did not begin until January last year. It is expected that the development and testing of components will be finished this summer, after which about 1½ years will be required for construction.

The reactor has been designed solely for research purposes, and is intended to operate at a heat energy output equivalent to 160 kW. Short-lived isotopes such as Na<sup>24</sup> could be produced in the reactor, which would be a particular advantage in the case of centres a long distance away from the usual production facilities, but the major uses are expected to be the study of materials subjected to neutron bombardment either in the pile itself or outside and for experiments in reactor development and for training scientific and engineering personnel in reactor operation. The total cost of the reactor and necessary buildings would be in the region of \$1,500,000, a sum perhaps within the means of some universities and large industrial organisations.

## Reactor construction

The reactor as designed is octagonal in shape, and when completed would measure 11 ft. high and 19 ft. wide and weigh approximately 450 tons. The core is a hexagonal prism, 4½ ft. by 4½ ft., consisting of enriched uranium sealed in a gas-tight aluminium tank and surrounded by a graphite reflector weighing 34 tons and about 30 in. thick. A shield of 6-in. steel and 3-ft. concrete is placed on the top and bottom of the reflector and on six sides of the octagon to protect operating personnel, while on the other two reactor faces the reflector graphite is extended as a thermal column beyond a bismuth shield, intended to stop gamma-rays but not neutrons. A removable cadmium and lead shield at these two faces permits access to the thermal column, and the shield at the top of the core is also removable, so that the core may be lifted out and replaced with the aid of an overhead crane.

Experimental holes for sample irradiation include one that is of 6-in. diam. and 15 of 2-in. diam., and the design may



Cut-away view of the North American low power research reactor. A, top shield; B, rod operating mechanism; C, graphite reflector; D, instrument hole; E, core; F, universal experimental hole; G, control-safety rod sheath; H, rod stop; I, bottom shield; J, heavy water out; K, heavy water in; L, bismuth; M, graphite thermal column; N, lead shield; O, concrete shield; P, multiple specimen irradiation tube; Q, pneumatic tube terminal.

include up to six vertical pneumatic tubes in the reflector and up to six multiple-specimen irradiation tubes, each of which will hold about 35 samples. The reactor is cooled with heavy water in a closed circuit, which in turn is cooled by ordinary water.

The reactor would be controlled from a console desk in the control room overlooking the main floor, so that experiments in progress can be watched. The power output of the pile is held within design limits

by adjusting the position of cadmium control rods projecting into the core, automatic devices tripping these rods in the event of any rapid rise in power. The reactor has also been designed to have a negative temperature coefficient of reactivity, so that neutron production would decrease as the reactor temperature rose. The maximum heat energy output of the pile would be equivalent to 200 kW, but at its designed output of 160 kW it could operate for ten years before the removal of the spent fuel would be necessary, thus obviating any need for the frequent disposal of fission products.

It is believed that construction of a reactor of this type has not as yet been authorised, but it is hoped that interest will be shown by industry and research organisations. As a reactor designed specifically for peaceful atomic energy research, the North American design should receive the support it deserves.

## Contributions

THE EDITOR welcomes practical articles and notes on chemical engineering and industrial chemical subjects with a view to publication. All contributions, which should be fully illustrated whenever possible, are carefully considered. A preliminary letter or synopsis is advisable. Address such material to THE EDITOR, CHEMICAL & PROCESS ENGINEERING, 17 Stratford Place, London, W.1.

# Dust Removal from Boiler Flue Gases

By G. C. Goodwin, A.C.G.I., M.I.E.S.

It is now almost universal practice to provide factories and power stations with some means of removing dust from the waste gases of their boiler plants. As is pointed out in the following paper,\* there are three usual methods of dust precipitation: gas washing, centrifugal collection and electrostatic precipitation. The author describes these systems briefly before discussing methods of testing the performance of such equipment.

**A**LTHOUGH the problem of removing dust or fly-ash particles from boiler flue gases is one that goes back a good many years, it is really only during the last 15 or 20 years that it has been seriously tackled and that progress has been made. On the one hand, modern standards demand that atmospheric pollution shall be reduced to the minimum and, on the other, higher boiler ratings, the increasing use of pulverised fuel firing and the use of low-grade fuels has increased the amount of dust in the waste gases. The earliest apparatus was only expected to entrap the coarsest or grit particles. The modern tendency is to class all solid particles in the waste gases as dust, and the collecting plant is expected to show a high efficiency when handling all the solid matter. At first sight, this may appear to be simply a problem of more efficient design on the part of the manufacturer, but there are a number of difficulties quite outside his control, all of which influence the final result.

Dust precipitation is usually done in one of three main ways: (1) by washing the gas, (2) by the use of centrifugal collectors and (3) by electrostatic precipitation.

## Gas washing

In general, gas washing is only installed where it is required to eliminate the oxides of sulphur which are always present in the waste gases to a greater or lesser degree, as well as to entrap the solid particles. It is usual to remove the bulk of the dust with a centrifugal collector and to employ the washer to deal with the remainder of the dust and the sulphur oxides.

## Centrifugal collectors

There are many forms of centrifugal dust collectors, but all of them operate on the same general principles. The dust-laden gases are given a whirling motion, either by tangential entry into a more or less cylindrical vessel, or by vanes, when the centrifugal force diverts the dust particles to the boundaries of the vessel, where they are removed, while the cleansed gases leave by an outlet duct which is usually concentric with the cylindrical vessel. In some forms the separation takes place in a single vessel, while in others the concentrated dust is bled off from the first cylinder with sufficient gas to convey it,

and the final separation of the dust from the conveying gas takes place in a second collecting vessel.

## Electrostatic precipitators

Dust particles passing through a very intense electrostatic field become charged and are attracted to an electrode of opposite polarity. The voltages used are high, being of the order of 30,000 to 60,000 V., and are usually obtained by rectifying a high-voltage alternating current. It has been suggested that by using a centrifugal collector as a pre-cleaner before an electrostatic precipitator, the former will reduce the dust burden so that the latter is then well able to take care of the extremely fine particles.

## Dust-collector performance

The user of dust-collecting equipment is chiefly concerned with the amount of dust being discharged from his chimney, whereas the maker of the plant is mainly interested in the amount and grading of the dust with which the collector has to deal. Unfortunately, developments in the design of various forms of firing a boiler have progressed faster than the ability to provide data as to the amount of the dust burden in the gases required to be cleansed and the nature of the dust to be removed. Much research will have to be undertaken before accurate information on the amount and nature of the dust burden to be anticipated with different coals and different methods of firing becomes available.

The development of a certain type of dust-collecting plant is largely dependent upon tests carried out in the manufacturer's laboratory, and these tests are usually run using cold air and a caught dust. In comparison with a site test, the laboratory test has the advantage of the comparative ease with which it is carried out. On the other hand, the dust used is caught dust which may not truly represent the actual dust to be extracted. Further, with some types, it is necessary to use a model of the collector or a few units from the full-size collector. Finally, cold air has a different density and viscosity to flue gases under site conditions.

The degree of uncertainty introduced in the laboratory tests by these discrepancies sometimes necessitates a full-scale test on the plant. Site tests, of course, are expensive and, although every care may be taken to preserve accuracy, the conditions are not as fully under control

as in the laboratory. The difficulty lies in obtaining a representative sample of dust fed into or out of the collector. This is generally obtained by traversing the cross-section of the flue at a suitable position by a probe through which gas is exhausted at a suitable rate. If the distribution of dust in the waste gases were uniform and the temperature constant over the cross-section of the duct or flue and, if the average direction of the gas flow at the point of sampling were parallel to the walls of the flue, most of the difficulties in site testing would disappear, but, unfortunately, since boiler plants are built for other purposes than those of collector testing, even an approach to ideal conditions for gas sampling is seldom attainable.

If possible, the sampling position should be in a run of duct with a considerable length of straight on both the upstream and downstream sides. Some authorities recommend that a special vertical test duct should be inserted in the flue, but the resulting dislocation on the operation of the boiler plant would usually prevent this refinement from being adopted. Having chosen the sampling station with a view to obtaining as nearly as possible the ideal conditions described above, arrangements must be made for sampling at equal intervals under like conditions.

Having withdrawn the representative sample of the flue gas, it is then necessary to extract the dust from it. The earlier types of test apparatus made use of paper, or alundum thimbles or flannel bags. The first two have the advantage of being compact and can frequently be located immediately behind the probe, but they limit the volume of gas that can be sampled. They also have other disadvantages. The ideal arrangement is one which has a relatively low pressure drop, which will permit a relatively large sample to be withdrawn and one in which the resistance does not build up unduly as the test proceeds, due to the resistance offered by the dust deposited in the filter. An arrangement which fulfils these conditions admirably is one in which the bulk of the dust in the gas sample is extracted by means of a cyclone and the final separation is effected by a filter of glass wool or other substance.

Returning to the quantities which are required for a site test, it is only in a very few cases that satisfactory sampling positions can be found both before and after a collector, but, in such ideal cases, it is only necessary to sample at these two

\* Summarised from 'Dust Precipitation,' read before the Manchester Association of Engineers on January 25, 1952.

points and to calculate from the results the amount of dust per pound or cubic foot of gas at the inlet and outlet of the collector. The efficiency of the collector, which is what is generally required, is then at once apparent.

In the majority of cases, a more or less satisfactory position for sampling can only be found either on the inlet or the outlet of the collector and, in this case, resort has to be made to the actual weighing of the dust brought down by the collector during the test.

If sampling is carried out at one side, either the inlet or outlet, then the total

amount of dust entering or leaving the collector during the test can be obtained from the dust burden in the sampled gas and the volume of gas passing the collector. Either of these values, together with the total amount of dust caught by the collector, will give the efficiency.

More accurate results will be obtained if the sampling is carried out at the outlet of the collector rather than at the inlet and for this reason it is preferable, wherever possible, to arrange for the sampling to be done on the outlet side.

The continued and ever-increasing demand for higher efficiency in the elimina-

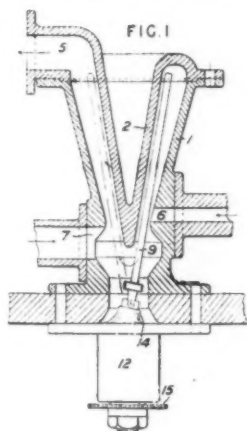
tion of solids from the waste gases of power station chimneys has caused the whole problem to become somewhat unbalanced, since the degree of elimination required is almost beyond the capabilities of existing methods of measurement. Normal requirements in respect of industrial undertakings are not quite so stringent, but as regulations governing atmospheric pollution are now being enforced more rigidly, more attention will have to be paid to the design of dust elimination equipment if the cost, which rises very steeply with the efficiency of the collection, is not to become exorbitant.

## Chemical Engineering Invention

### MONTHLY SUMMARY OF PATENT CLAIMS

#### Emulsifying apparatus

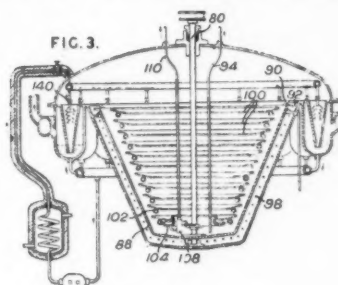
**A**n apparatus for mixing fluids in flow or for preparing foam comprises a vessel having walls defining a cone-shaped annular chamber having a fluid inlet near the narrow end, a fluid outlet near the wide end, and means for imparting turbulent gyratory movement to fluids in the chamber. Separate fluids, e.g. a liquid and a gas, are introduced through inlets 6, 7, (Fig. 1) and pass through the annular space between conical members 1, 2, to be agitated by a stirrer 9. The mixture is discharged through an outlet 5. The stirrer 9 is moved in a conical path around the annular chamber by mechanism contained in a casing 12. The discharged mixture may be divided into two streams and pumped back to the apparatus, one stream to each inlet. In a modified con-



struction, the inlets 6, 7, may be replaced by a single inlet for the fluids to be treated. In a further alternative construction, liquids only are introduced through the inlets 6, 7, a separate inlet being provided for a gas.—610,974, W. Binns, Bintex Ltd. and International Latex Processes Ltd.

#### High vacuum distillation

Liquid to be distilled is fed through pipe 94 and spreads over a vaporising plate 88 rotated by shaft 80 and heated by element 98. The coils 102 cool condens-



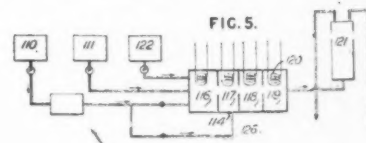
ing collars 100, and the condensate is collected in the gutter 104 and delivered to the pipe 110 by the pump 108. Undistilled residue is thrown from the periphery 90 and collected in the gutter 92. A high vacuum is maintained in the apparatus by a number of ejectors 140 connected in parallel and each connected to a backing pump.—608,249, Distillation Products Inc.

#### Continuous saponification

An aqueous saponifying reagent and a fat are passed continuously through a series of mixing compartments maintained at a saponifying temperature, and the conditions of saponification are adapted in each compartment to maintain a saponified mixture in which the degree of saponification of the available fatty acid is either below 70 or above 85%. The degree of saponification in each compartment is determined, for example, by the temperature and time during which the reagents are together in the compartment. The saponification may be carried out so that less than 70% of the available fatty acid is saponified in the first compartment, and in

a subsequent compartment the degree of saponification is raised to over 85%. Alternatively, saponification in excess of 85% may be effected in the first compartment. In order to obtain effective utilisation of the aqueous saponifying reagent, a further quantity of fat may be fed into a compartment down-stream from the compartment in which saponification has proceeded to an extent in excess of 85%, provided that the degree of saponification is not brought within the range 70 to 85%.

For example, in Fig. 5, fat from container 110 is passed in confluence with lye from container 111 into the first compartment 116 of the mixer 114 provided with a heating coil 120, wherein saponification occurs to an extent in excess of 85% of the available fatty acid. Brine may also be fed into the compartment 116 from container 122 in order to grain the soap as it is formed. Coils 120 in compartments 117, 118 and 119 may be used to cool the soap before it enters the centrifuge 121. A further quantity of fat may be introduced into 117 through pipeline 126 to utilise excess lye contained in the soap, but



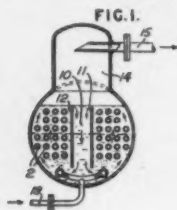
insufficient to produce a mixture in which less than 85% of the available fatty acid is saponified.

In a modification, the fat fed from container 110 may be a mixture of soap and fat derived from the first stage of a multi-stage process in which saponification has proceeded to an extent corresponding to less than 70% utilisation of the available fatty acid. Specifications 549,386 and 549,387 are referred to.—608,943, Sharples Corpn.



## Bulk evaporators

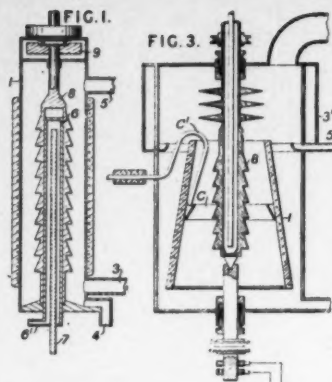
In an evaporation in which the medium to be cooled flows through horizontal tubes 2 surrounded by the refrigerant which is then heated and evaporated, a space 10 is provided between two banks of the tubes 2 extending over the whole length of the evaporator, open at the top and bottom to cause a flow of refrigerant liquid upwardly over the tubes 2 and downwardly through the space 10. The space 10 is formed between walls 11, 12, and the level of the liquid is arranged to be always above the tops of these walls. The refrigerant is fed through a pipe 13



branched inside the casing into two extended spray pipes situated beneath the tubes 2. The refrigerant vapour collects in a dome 14 and passes out through a pipe 15. The flow of refrigerant removes gas bubbles from the surface of the tubes 2. The medium to be cooled passes in series through the two banks of tubes 2.—609,986, Escher Wyss Maschinenfabriken Akt.-Ges.

## Fractional distillation

Fractional distillation is carried out by establishing an unobstructed annular stream of the vapour mixture between a single rotating condensing surface and an enclosing evaporating surface, whereby condensate formed on the condensing surface is continually flung across the vapour stream and into contact with the evaporating surface. By these means, high vapour velocities can be used without entrainment of condensate and without causing a large pressure gradient across the fractionator. Fig. 1 shows an embodiment suitable for operation at absolute pressures between 1 mm. and 40 mm. It comprises an electrically heated casing 1 and a stationary tube 6, supplied with cooling fluid through conduits 7 and 6', upon which rotates, by means of magnetically driven armature 9, a sleeve 8 in the form of a series of truncated cones. The vapour to be fractionated is introduced at inlet 3, and bottom product is returned to the still through line 4. The overhead product is withdrawn through outlet 5 which is connected to a final condenser and a vacuum pump. Fig. 3 shows an embodiment in which the electrically heated evaporator 1 rotates as well as the condenser 8. The liquid to be distilled is fed through electrically heated pipe C' into perforated, annular distributor C. The overhead vapours are



condensed by upper cooling jacket 3', and the condensate is removed through outlet 5. A non-condensable gas may be injected near the base of the fractionator to provide a carrier to take the vaporised material overhead. The use of high vacua, i.e. pressures below 1 mm., is referred to.—614,388, Anglo-Iranian Oil Co. Ltd. and M. E. Kelly.

## Drying of Solids

THE capillary theory does not altogether account for the behaviour of finely divided granular solids during drying, since it does not allow for the peculiar behaviour of water when it is present as adsorbed films on the solids. The mechanism of the drying of solids has been the subject of a series of papers before the Institution of Chemical Engineers by Prof. D. M. Newitt, F.R.S., and Mr. M. Coleman. In their third paper, recently presented, their studies are extended to a more complex fine solid, china clay.

Prof. Newitt, who read the paper, pointed out that when a plaster clay-water mix is permitted to dry, it undergoes shrinkage during the constant-rate period, then during the falling-rate period the pure water is removed. Two principal theories have been presented to explain its behaviour, the capillary theory, and the more widely accepted 'osmotic imbibition' theory.

Water was expelled mechanically from prepared samples by the application of high pressures, and the driving forces or 'suction potentials' were measured. Measurements were also made on samples in which the surface tension of the water had been artificially lowered. As shrinkage water is expelled, suction potentials increase, until a point is reached corresponding to the critical moisture content, and which differs according to the method of preparation of the clay sample. From the nature of the results it is evident that capillarity is not the origin of suction potentials in clay.

The effect of the thickness of the clay bed on the rate of drying has been studied,

and the desirability of drying clay in as thin a bed as possible is clearly shown by these results. Striking differences have been found between the moisture distribution in drying plastic clay and in drying clay slurries. The experimental work showed that during the falling rate period of drying after the critical moisture content had been passed, the principal influences are (1) the immobility of the remaining moisture which obliges it to vaporise *in situ*; (2) the progressively decreasing vapour pressure of the remaining moisture; and (3) resistance to vapour flow through the fine-pored structure of the body, added to that of the vapour film.

## New standards

**Toroidal sealing rings.** A British Standard, B.S. 1806: 1951, for dimensions of toroidal sealing rings has been prepared at the request of the Ministry of Defence. It applies to the dimensions of toroidal sealing rings, sometimes referred to as 'O' seals, which are used as packings for reciprocating mechanisms and as gaskets for static purposes for light and medium general engineering applications. These sealing rings are not intended for use at pressures in excess of 1,500 p.s.i. For use as packings on reciprocating mechanisms 73 sizes are specified in the range of  $\frac{1}{8}$  in. to 8 in. and for static purposes 96 sizes in the range of  $\frac{1}{8}$  in. to 15 $\frac{1}{2}$  in. This latter range includes the 73 sizes intended for reciprocating mechanisms.

Recommendations are also included for the recesses into which these rings are fitted, but the standard emphasises the necessity for close collaboration between the designer and the manufacturer of sealing rings to ensure satisfactory operation and service. The standard costs 2s. 6d., post free.

**Automatic pipettes.** B.S. 1583, 'one-mark bulb pipettes,' published in 1950, specified a range of 16 sizes, being those most in demand for general laboratory use. A specification (B.S. 1132: 1952) has now been prepared, on similar lines, for automatic pipettes. This specifies eight sizes of pipette from 2 ml. to 100 ml. capacity. It gives full dimensions but, in accordance with current practice in British Standards for laboratory glassware, lists only the essential ones as mandatory, the remainder being given for the guidance of manufacturers. The design includes an overflow cup, and a special alternative type is described for work in which the liquid must be kept sterile. It includes standard methods for the determination of capacity and delivery time, and tolerances for both. An appendix gives the sizes of glass tubing recommended for the manufacture of these pipettes. This standard costs 2s., post free.

Both the above are available from the British Standards Institution.

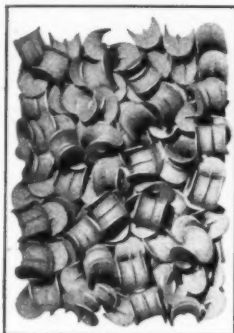
# Plant and Equipment

## Tower fillings

More than 700 cu. ft. of tower fillings are produced and supplied each week by F. Weinreb & Co. Ltd., of London, who have specialised in these chemical engineering accessories for nearly 30 years. This figure takes into account the considerable range of sizes, starting with 5-m.m. Berl saddles and 1-in. Raschig rings up to 4-in. or even larger rings. These tower fillings are made in either laboratory porcelain in certain smaller sizes, or in white, iron-free, acid- and heat-shock-resisting stoneware containing the minimum of acid-soluble salts. These characteristics are of the greatest importance to the users of these fillings, either in distilling columns or absorption towers, stripping and cooling towers.

Although the manufacturing process is highly mechanised, aiming at a maximum output, individual requirements can be met.

The fillings are made of a material which, in addition to the qualities mentioned, has good mechanical strength, so that they can support heavy superimposed loads formed by the upper layers of smaller rings of saddles (which are heavier per cu. ft.), or of catalyst carriers. The non-porous fillings, as used in essence stills, or



Berl saddles.

working at 600°C., can be cleaned repeatedly.

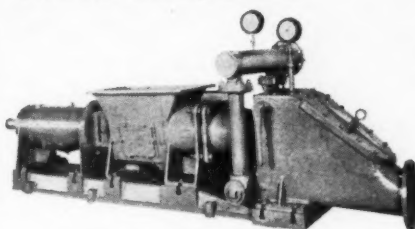
Porous fillings can be supplied for use in special reactions, as well as carbon and glass types.

## Pneumatic conveying system

The Fuller-Kinyon pump has been employed for many years for the low-pressure conveying of many types of dry pulverised materials. This pneumatic conveying system is particularly suitable for the handling of such materials as fuller's earth, clays, soda ash, powdered aspirin, gypsum, carbon black, various starches, etc. The pump operates successfully for conveying distances from 50 to 1,000 ft., and various sizes of equipment are supplied for pumping up to a rate of 120 tons/hr.

Compressed air is normally supplied by a rotary compressor directly connected to the Fuller-Kinyon pump. The pressure in the conveying system varies according to the tonnage and conveying distance involved, but generally speaking line pressures vary between 5 and 20 lb./sq. in. An advantage of using such a conveying system is that it lends itself admirably to extensions, and particularly when conveying problems have to be overcome in existing plants, since the conveying line can be installed without alteration to any existing building or plant.

Maintenance costs are negligible as the working parts of the pump are made of special wear-resisting materials.



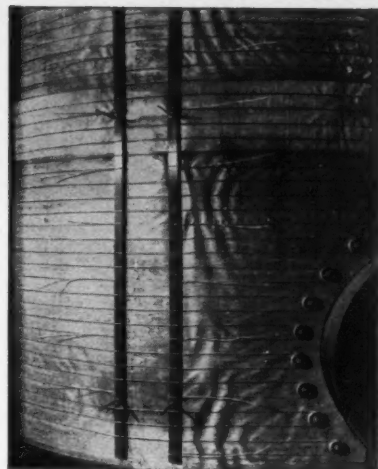
Conveying system for dry, pulverised materials.

The system, being a totally enclosed one, limits dust nuisance which causes diseases such as silicosis. The spent air is normally discharged to atmosphere through large diameter vent stacks in the receiving hoppers, or alternatively by means of cylindrical stocking filters. Further, the materials are not segregated according to size or weight.

The system is especially suitable for handling combustibles such as starch and coke dust, eliminating fire and explosion hazards. The quantity of air required for aeration and conveying of the material is less than 1% of that necessary to support combustion. This pump is supplied by Constantin (Engineers) Ltd.

## Sugar vats heated with soil warming equipment

An unusual application of soil warming equipment has come to light in a Hampshire brewery. Three years ago Courage & Co. Ltd., brewers, were faced with the problem of maintaining the temperature of sugar syrup in two 60-barrel capacity tanks at their Alton Brewery at 110°F., with a minimum ambient temperature of 40°F. They approached the General Electric Co. Ltd., and suggested that these tanks, which are 5 ft. 6 in. in diameter and 8 ft. high, should be heated by elements from the outside. This has been done by means of 15 s.w.g. galvanised soil heating wire laid on each tank in 12 sections of four turns each. The sections are connected in parallel and fed from a 2.5 kVA soil heat-



The 15 s.w.g. galvanised soil-heating wire laid on a sugar vat encased in glass fibre.

ing transformer on a 15 V secondary winding. They are controlled by means of a suitable calibrated long-stemmed thermostat inserted into the liquid near the bottom of the tank.

To support the bus-bars and wire spacers, studs were welded to the exterior of the tank at suitable intervals. The tank was then served overall with a layer of glass fibre cloth impregnated with special shellac 'dope,' wire spacers suitably slotted were placed in position and the wire was wound on. Another layer of glass fibre cloth was then laid overall and the terminations of the windings brought out to the bus-bars held on the studs. Finally, two layers of cork slab lagging 1½ in. thick were put over the whole installation, and held in position by a sheet metal cover drawn tight by suitable tensioners. The equipment has worked satisfactorily since it was installed two years ago.

## Coating underground pipes in situ

The corrosion of buried pipelines has long been a serious problem for public authorities, collieries, gas undertakings, etc., and the conditions to which such pipelines are subjected are usually more severe than is realised. The preservative properties of bitumen have been known for centuries but research carried out by Mercol Products Ltd. has shown that for the internal coating of underground pipe-work, it is necessary to add other resistant materials to a bituminous base to counteract specific conditions of corrosion and scale formation. A bituminous based coating for underground pipes was evolved, the formula for which is varied to suit the particular conditions of service. Further research was then devoted to finding the best means of mechanically applying the product, *Mercoloid*, this problem of coating by remote control having baffled many engineers in the past.

The basic principle of the Mercol positive pressure system lies in the keeping

of a reservoir of *Mercoloid* in high pressure contact with the walls of a pipe for a sufficient length of time to enable the compressed viscous material to expel all residual moisture and secure a positive grip upon the walls of the pipe. The reservoir, or pressure chamber, is formed by a pair of sliding pistons connected in tandem, the space between the pistons being primed with *Mercoloid*.

The pistons are pulled through the pipe a number of times, to and fro. Some idea of the pressure applied will be conveyed when it is realised that it requires a 10 h.p. winch at each end of the pipe section, with the engines running in low gear, to draw the apparatus against the compression created by the 'concertina' action of the pistons and the 'drag' of the dense mass of viscous *Mercoloid*. The capacity of the reservoir is adjustable to suit the length and diameter of the pipe being cleaned. Pressure is automatically maintained during the traverse of the pistons in either direction, this reversible 'concertina' feature being embodied in the Mercol patent.

This process is applied after the pipe has been descaled and cleaned, and given a final scouring with rotary wire brushes.

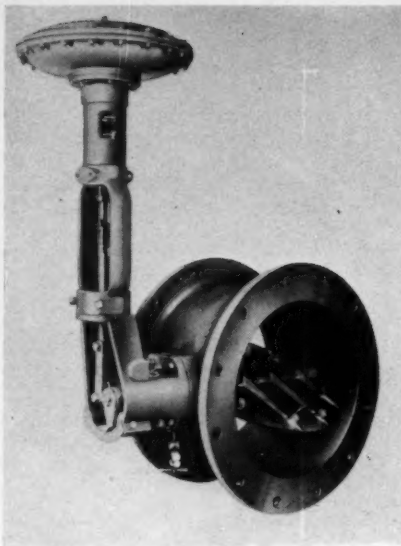
#### Photoelectric control devices

A new industrial smoke density indicator which operates photoelectrically, actuates an alarm or controls in the event of excessive smoke discharge from power station or factory chimneys. This unit can also be used as a fire alarm and is proving useful in protecting products liable to spontaneous combustion. By slight modification this unit can be employed for checking turbidity of liquids, and in an adapted form, is installed in grass and grain drying plant as a means of protection against contamination from fuel flames.

The photoelectric principle is also used in the *Flamestat* unit developed as a precaution against flame failure when using coal gas or oil as a fuel. The unit automatically cuts off the fuel supply should the burner flame go out; likewise, it is impossible for the fuel to be turned on again until the lighting torch is actually in position against the burner.

Similar units are used to control temperatures. One application is the employment of a unit which is operated by the infra-red rays emitted from an electrically heated bar, the unit in this case switching off the current when the pre-set temperature is reached. This application is also used to control welding temperature on automatic chain making machines.

A somewhat different device is a photoelectric unit which counts moving articles at speeds up to 22,000 per min.; it is also possible to count in batches of any required number; when the pre-set number has passed the unit, the machine can be automatically stopped or an audible warning given. These units are supplied by Radiovisor Parent Ltd.



Pneumatic valves for the control of liquids and gases.

#### Pneumatically-operated motor butterfly valves

To meet the need for pneumatically-operated valves for the control of liquids and gases under relatively low static pressures, Fisher Governor Co. Ltd. have added a range of butterfly valves to the series of ported valves, pressure regulators and other specialities for automatic control, now made by the company in England.

The butterfly valves are available in sizes from 4 to 24 in., and control the flow of fluid by a disc on a rotary shaft linked in a simple manner to a pneumatically-operated diaphragm motor of the same type as used on the well known Fisher ported valves. The discs are shaped and ribbed for maximum strength and offer minimum resistance to fluid flow. In the closed position the clearance area between the disc and body does not exceed 0.5% of the total pipe area.

By the use of a rugged crank and lever with accurately ground pins, lost motion in the operating mechanism is reduced to a negligible amount whilst reliability is ensured.

Standard diaphragm pressure range is 3 to 15 lb./sq. in. and for automatic pressure regulation the Fisher Wizard pilot can be mounted integral with the diaphragm casing. The Fisher Positrol valve positioner can also be fitted if required.

Typical applications for these valves are for coal and producer gases for the gas industry, for large low-pressure gas lines in steel works, oil refineries and chemical plants and low-pressure exhaust steam controls.

**For further information on new plant and equipment, please complete the coupon on page 208.**

#### Pentachlorophenol against wood termites

In the article in the March issue entitled 'Modern Wood Preservatives' the following statement is made under the section headed 'Protection of Green Timber':

'Chlorinated phenols have been tested in the laboratory and in the field. Pentachlorophenol, an excellent fungicide, has not proved effective against the wood termites.'

This is a condensation of a passage from H. Allott's paper, 'Results of Research on the Chemical Production of Green Timbers,' and we regret that in making the summary the sense of this passage was distorted resulting in the mis-statement that pentachlorophenol is not effective against wood termites. The original sentence reads:

'The chlorinated phenols, and particularly pentachlorophenol, an excellent fungicide, the efficacy of which against termites is well known, have not shown themselves to be very effective in this case.'

The meaning, obviously, is quite different from our summary.

M. Allott was referring to a specific problem, the protection of freshly felled timber of high moisture content against attack by insects commonly known as ambrosia or pinhole borers, shot-hole borers and long-horned beetles. That pentachlorophenol has not been used to any great degree in controlling these insects in freshly felled logs is due mainly to the difficulty in applying oil-soluble preservatives to timber with a high moisture content and not to any lack of toxicity of pentachlorophenol towards these insects.

Pinhole borers attack only freshly felled timber and none of the chemicals normally regarded as wood preservatives is effective in preventing their attack. Shot-hole borers and long-horned beetles are much better known for the damage they cause to seasoned timber, and Mr. Ira Hatfield, the American authority on wood preservation, in a paper read before the Forest Products Research Society entitled 'How to Recognise and Control Powder Post Beetles in Wood' reported as follows:

'... actual poisoning of the wood is necessary if *Bostrychids* (shot-hole borers) are to be kept out of wood. The most useful chemical found to date for this purpose is pentachlorophenol applied in a suitable petroleum oil carrier; and

'In the case of long-horned beetles like the "flat oak" and "old house" borers that attack seasoned wood, it has been observed that these beetles are controlled by a preservative treatment such as 5% pentachlorophenol in oil.'

In summarising the paper he stated:

'It might seem that the control measures given for each group of insects made reference too often to the use of pentachlorophenol in oil. If there were other usable products that would impart prolonged protection they would have been mentioned.'



# World News

## GREAT BRITAIN

### N.R.D.C. may help production of rare chemicals

The possibility of assisting the production of small quantities of new chemicals for test purposes is being studied by the National Research Development Corp., the Government-sponsored organisation for promoting the development of inventions. In a talk on the corporation given at the Royal Society of Arts, London, its managing director, the Earl of Halsbury, F.R.I.C., stated that there are inadequate facilities for producing, for clinical and other tests, small quantities of chemicals of possible use in medicine or as insecticides or veterinary products. The matter has the attention of the corporation with the object of finding a solution, but early results are unlikely.

The corporation is also interesting itself in the production of highly specialised scientific instruments and is in touch with the British Scientific Instruments Research Association and the Scientific Instrument Manufacturers' Association to decide what assistance can be given.

### Industrial gases and the Monopolies Commission

In Parliament on March 4, Mr. Baker White, Conservative member, asked the President of the Board of Trade whether he would cause the Monopolies Commission to investigate the practices of the British Oxygen Co. Ltd. In a written reply, Mr. Henry Strauss, the Parliamentary Secretary, stated that the Minister will bear in mind the supply of industrial gases when further references to the Monopolies Commission are selected.

### Output and allocation of steel

The Minister of Supply was recently asked in Parliament what steps he was taking to improve the output of steel. He replied that the expansion of the output of steel depended primarily on increased supplies of raw materials. His Ministry maintained very close touch with the industry in this matter and every effort was being made to improve the position.

### Chemical works safety conference

The Association of British Chemical Manufacturers is holding a chemical works safety conference at the Palace Hotel, Buxton, Derbyshire, from Friday to Sunday, May 2-4, 1952. Papers are to be read on the following subjects: 'Safety in the Laboratory and on the Plant,' by Dr. L. J. Burrage, I.C.I. Ltd.; 'Toxic Dusts in Industry,' by J. S. Evans, Federation of British Industries; 'The Relation between Plant Strength and Dust Explosion Relief,' by Dr. D. Matheson, Ministry of Labour; 'Safety in Plant Maintenance,' by C. A. Beaton, Monsanto Chemicals Ltd.; 'The

Safe Transport and Storage of Chemicals,' by the Works Safety Committee of the A.B.C.M. The chairman of the conference will be Mr. J. Davidson Pratt, director of the association.

The fee per delegate is £8 8s., and full details can be obtained from the A.B.C.M., 166 Piccadilly, London, W.1.

### Co-operation in gas research

An agreement on research policy has been reached between the Society of British Gas Industries and the Gas Council. In addition to collaboration with individual member firms of the society, which has been the practice for many years, the agreement provides for a Joint Consultative Committee on Research of the Gas Council and the society which will be free to discuss any questions of research suggested by either party, and to remit these matters for study and prosecution.

### Heavy chemicals productivity team

A heavy chemicals productivity team left on March 6 for a six-week tour of the U.S.A. to study productivity in the American chemical industry. The team is led by Mr. J. G. Moore (deputy works manager, General Chemicals Division, I.C.I.). Other members of the team are: *Supervisory and technical level*—Dr. E. Brookman (general works manager, Plastics Division, I.C.I. Ltd.), Mr. G. Clayton (personnel manager, Hardman & Holden Ltd.), Mr. A. Gait (operation planning specialist, Petrochemicals Ltd.), Mr. G. Maddra (head of Technical Development Department, Peter Spence & Sons Ltd.), Mr. J. Sommerville (plant manager, British Titan Products Co. Ltd.), Mr. S. Warr (works manager, Peter Spence & Sons Ltd.), Mr. E. Weekes (area manager (production), Monsanto Chemicals Ltd.), Mr. A. Wells (director (production and factory), A. Boake Roberts & Co. Ltd.); *Workshop level*—Mr. R. Amer (leading hand, Brotherton & Co. Ltd.), Mr. J. Crooks (process worker, British Titan Products Co. Ltd.), Mr. E. Hazledine (chief operator, Petrochemicals Ltd.), Mr. G. Leather (boilermaker, General Chemicals Division, I.C.I.), Mr. J. Malloch (chargehand, Hardman & Holden Ltd.), Mr. J. Walker (stillman, Hull Distillery Co. Ltd.), Mr. M. Scott (general worker, Billingham Division, I.C.I.). The team secretary is Mr. D. Ladham (works manager, British Industrial Solvents Ltd.).

Visits will be paid to manufacturing plants, mainly in the southern States, including:

American Cyanamid & Chemical Corp., Stamford, Connecticut;  
Monsanto Chemical Co., St. Louis, Missouri (Phosphate Division);  
Commercial Solvents, Sterlington, Louisiana;

Celanese Corp. of America, Chemcol, Texas;

Shell Chemical Co., Houston, Texas;  
Down Chemical Co., Freeport, Texas; and

Diamond Alkali Co., Houston, Texas.

The team was invited to attend the Convention of the American Institute of Chemical Engineers at Atlanta, Georgia, on March 16-19.

A report embodying findings and recommendations considered applicable in the interests of efficiency and productivity in Britain will in due course be published in the series issued by the Anglo-American Council on Productivity.

### Dearer iron and steel

The Minister of Supply has increased the controlled maximum prices of iron and steel. This is due partly to the higher cost of imported steel and partly to higher costs at home. Typical examples of the price changes are:

	Revised basis price			Former basis price		
	£	s.	d.	£	s.	d.
Basic pig iron ..	12	10	0	11	15	6
Soft basic billets ..	25	4	6	21	11	6
Sheet and tinplate bars ..	25	3	6	21	16	0
Heavy sections ..	27	17	0	23	15	6
Plates (N.E. coast, etc.) ..	29	14	0	25	6	6
Light sections ..	31	5	6	27	1	0
Bright steel bars ..	42	7	3	37	6	9

Following these increases the controlled maximum prices of nuts, bolts, etc., have also been raised.

It will be recalled that it was due to disagreement with the Minister on the need to raise iron and steel prices that Mr. Steven J. L. Hardie resigned the chairmanship of the Iron & Steel Corp. of Great Britain in February. Before this appointment Mr. Hardie was chairman of the British Oxygen Co. Ltd.

### Petrochemicals' new plants

In his statement with the accounts for the year to June 30, 1951, Sir Robert Renwick, chairman of Petrochemicals Ltd., states that all the company's plants are now operating, the majority at, or better than, the rated capacity. The average naphtha throughput during the last six months has been at the rate of 80,000 tons p.a., which is in excess of the rated capacity. The gas separation plant, which initially gave operational trouble, has in recent months been performing very satisfactorily. However, since September 1951, it has been operating only on No. 2 cold box in order to permit modifications to No. 1 cold box. These modifications should be completed by the end of March 1952, but so long as one cold box is out of action, the firm is in a position where an extremely complicated plant—upon which much of the revenue of the company depends—is being operated without standby capacity.

The continuing shortage of chlorine,

which the company have to buy from outside sources, has not permitted them to take full advantage of the improved performance of the plants and, as a result, the conversion of ethylene to ethylene oxide has been below capacity. The chlorine position is a prime factor in the company's revenue position, and more favourable results would have been produced had they been able to obtain all desired supplies. It is now hoped that during the first quarter of 1952 deliveries of chlorine will reach total requirements, with the consequent improvement in the company's revenue position.

The associated and subsidiary companies have continued to make satisfactory progress. The arrangements made to supply the total requirements of Styrene Products Ltd. for monomeric styrene by imports from abroad have continued to work satisfactorily, and the technical performance of the Styrene Products plant has been excellent and profitable. Styrene Co-Polymers Ltd., which manufactures various paint and varnish media, based partly on a distillate supplied by the company, has steadily increased its production and has been earning profits since July 1, 1951—the current date of its financial year. Oxirane Ltd., which acts as a sales and development organisation for part of the range of ethylene derivatives, has continued to develop favourably, although its progress has been inevitably retarded by the chlorine shortage.

Turning to the accounts the chairman stated: 'It will be seen that in the consolidated profit and loss account there is an accumulated debit on the profit and loss account as at June 30, 1951, of £2,025,296, which is in accordance with the estimate in my last statement. Since July 1, 1951, the beginning of its current financial year, the current revenue expenditure (excluding interest) has been covered by current income, but there will inevitably be a further loss during the current financial year after making provision for depreciation and interest on the various loans which are outstanding.'

#### Obituary

We regret to announce the death of Mr. Herbert Dowell, Technical Director of Foster, Yates and Thom Ltd., engineers, of Blackburn, on March 4 after a long illness. Mr. Dowell was first associated with the company's predecessors in 1897.

#### Duke of Edinburgh Hon. F.R.I.C.

The Duke of Edinburgh has been elected the first Honorary Fellow of the Royal Institute of Chemistry. The Council intends shortly to elect further Honorary Fellows, restricting the number to seven.

#### Change of address

The National Sulphuric Acid Association has moved to Piccadilly House, 33-37 Regent Street (Piccadilly Circus), London, S.W.1. Telephone numbers, Regent 1440 and 3561/2/3, remain unchanged.

#### Mond Nickel fellowships

To help alleviate the shortage of qualified metallurgists available to industry, the Mond Nickel Fellowships Committee are inviting applications for five fellowships of average value £750 each for 1952. Awards will be made to British candidates educated to university degree or similar standard, though not necessarily qualified in metallurgy, who wish to train in industrial establishments for a year. Full particulars and application forms, which must be submitted before June 1, 1952, can be obtained from Mond Nickel Fellowships Committee, 4 Grosvenor Gardens, London, S.W.1.

#### Chemist leads Scientific Advisory Council

Prof. A. R. Todd, Professor of Organic Chemistry, Cambridge University, has been appointed chairman of the Advisory Council on Scientific Policy by the Lord President of the Council. Prof. Todd succeeds Sir Henry Tizard, who retired at the end of last month. Prof. S. Zuckerman will continue as deputy chairman.

#### Personal

Mr. A. B. GRAHAM, manager of the Zenith Works of Henry Wiggin & Co. Ltd. at Thornliebank, Glasgow, has been elected to the Delegate Board of the company. He was born in Scotland in 1889 and educated at Hutcheson's Grammar School and the Royal Technical College, Glasgow. For the past 18 years he has been in charge of Wiggin's Glasgow works and recently qualified for membership of the Inco Quarter Century Club by virtue of his 25 years' service with associated companies of the International Nickel Co. of Canada Ltd.

#### Seaweed harvester

A harvesting machine is being developed to expand the seaweed chemical industry in Scotland. Speaking at Glasgow, Dr. Woodward, of the Institute of Seaweed Research, said two harvest conveyor units were being built, based on a prototype which proved successful in sub-littoral harvesting. They were also developing a cut-suck type of unit. Both the cutting and sucking problems had been solved, and they were now engaged on the task of co-ordinating the cutting and sucking motions to ensure a steady efficient flow of cut seaweed to the hold of the operating ship. Dr. Woodward outlined the years of detailed work which had been completed and the vast amount of data on Scottish seaweed resources which had been built up.

The exploitation of the chemical derivatives was a matter for the industry as a whole rather than that for the scientists at work on the seaweed project. They were hopeful that industry would take the various chemical derivatives and find uses for them in their industrial processes. Already a considerable amount of work had been done on these lines, but the scope remaining was immense.

#### H<sub>2</sub>SO<sub>4</sub> from anhydrite plant

A start has been made on the construction of the 150,000-ton-p.a. sulphuric acid plant being built by the United Sulphuric Acid Corp. Ltd. at Widnes, in which anhydrite will be used as the raw material. The corporation has been formed by 11 British chemical companies (see INTERNATIONAL CHEMICAL ENGINEERING, May 1951, p. 237). The plant will cost more than £4,000,000 and, when completed, will employ between 250 and 300.

#### Patent Office Library

The extended hours of opening of the Patent Office Library, 25 Southampton Buildings, Chancery Lane, London, W.C.2, will be continued. The hours are 10 a.m. to 9 p.m., Mondays to Fridays, and 10 a.m. to 5 p.m., Saturdays.

#### New industrial alcohols

Two new industrial alcohols of medium chain length, *Alphanol 79* and *Nonanol*, are being produced by Shell Chemicals Ltd. These products are made in the U.K. from raw materials available locally by the carbonylation reaction, also known as the OXO process. Among a variety of industrial uses for which they are recommended is the manufacture of surface-active agents. They may be used in the preparation of sulphates, phosphates and sulphosuccinates and for the provision of alkyl groups in quaternary ammonium surface-active compounds.

*Alphanol 79* is claimed to be a good anti-foaming agent, and both materials can be converted into aliphatic aldehydes for use in compounding floral type perfumes and fruit flavours.

The new alcohols are recommended for the manufacture of plasticisers. They may also be used in synthetic lubricating oils, lubricating oil additives and special oils, coupling agents, surface coatings and speciality resins.

#### ISRAEL

#### New rayon and cellulose plants

Investigations are being made by the Scientific Research Council into the commercial usefulness of Israeli cellulose-bearing plants. Surveys so far carried out by the Council showed an unexpectedly high cellulose content in a number of indigenous trees and plants, and it is thought that locally produced cellulose might be suitable for the American-Israel Paper Mills Ltd. due to start production in March 1953 and the rayon plant planned by the American and Israeli investors.

Oscar Kohorn & Co., of New York, have invested \$2,000,000 in the rayon plant, which will involve a total investment of \$5,000,000 and £1,000,000. Initially, the rayon plant will have to import cellulose, and it will be three or four years before local raw materials could be used.

The cellulose plant is still in the planning stage, which is being handled by the Ministry of Development and Agriculture.



## GERMANY

### Hydrogenation plants restarted

The Krupp-Kohlechemie works at Wanne-Eickel, Ruhr, formerly Krupp-Treibstoffwerk, recently resumed production in part of its Fischer-Tropsch plant. High-boiling-point hydrocarbons and solid paraffins will be produced chiefly. Three other hydrogenation plants, Glesenberg Benzin, Chemischewerke Wesseling, and Scholven Chemie A.G., have also resumed production.

A fifth company, Ruhröl GmbH., at Bottrop, near Essen, belonging to the Stinnes group, hopes to restart its hydrogenation plant shortly. Reconstruction of this plant began in 1948. Since the war Ruhröl has been engaged only in processing naphthalene. This year, its monthly production of phthalic acid is expected to be 500 tons, making the firm one of the biggest producers of phthalic acid in Europe.

### New anaesthetic

The Alcid chemical works in East Germany is reported to have produced a new anaesthetic, *Falicain*. It is claimed that exhaustive tests show it can be used like cocaine and novocaine.

### Sulphur from coal gas

The recovery of sulphur from coal gas represents the best method of meeting the current sulphur shortage in Western Europe, according to Dr. Hans Broche, manager of the Stinnes coal mines. On the latest reckoning, he said, it would be possible to obtain 182,000 tons of sulphur from 26,000,000 cu.m. of coke-oven gases with a sulphur content of 7 gm./cu.m. if all the coke-oven gases produced in Europe were subjected to a sulphur recovery process. A decisive step towards the recovery of elemental sulphur from this source had been taken in West Germany in the extraction of the sulphur-bearing gaseous masses. The production of pure sulphur by extraction processes had already become West Germany's greatest single source of sulphur.

One-half of the available sulphur was still being lost, however, by the utilisation of raw gas from which the sulphur-bearing gases had not been extracted. It was Dr. Broche's opinion that the numerous stages of purification and manufacture still possible with coal and coal by-products offered the best available means of making the Ruhr coal mines more profitable.

### Potash production expands

Plans to expand German potash production this year are being made to meet the strong demand both from home and overseas. Reconstruction of the Königshall and Hindenburg shafts, both operated by the Burbach concern, have been completed. A new potassium chloride plant is expected to begin producing this month and will process 24,000 cwt. of the daily output of 30,000 cwt. to be produced there. Some

## The Leonard Hill Technical Group

Articles published in some of our associated journals in the Leonard Hill Technical Group this month include:

**Manufacturing Chemist**—Treatment of Waste Waters from Penicillin Manufacture; Ion Exchange in Chemical Analysis; Castor Oil; Nitrites in Medical Practice; Progress Reports on Disinfectants, Cosmetics and Fertilisers.

**Petroleum**—Scottish Shale Oil, Part I; Oil in Turkey; Making Refinery Equipment.

**Food Manufacture**—World Progress in Fish Canning, Part II.

**Textile Industries**—Ethylene Oxide, Its Significance for the Textile Industry.

**Paint Manufacture**—Canadian Raw Materials for the Paint Industry; The Physical Characteristics of Titanium Pigments; Carbon Paper Manufacture.

**Atomics**—Design and Layout of Radiochemical Laboratories; Geiger Counter Developments.

**World Crops**—Development of Rotary Cultivation; Principles of Low-Volume Spraying.

**Muck-Shifter**—Lubrication of Earth Moving Machinery.

**Building Digest**—The Plan of the Kuwait Oil Refinery Town.

D.M. 20,000,000 have been invested in this project.

The Hildesia shaft, belonging to the Wintershall concern, is now ready to resume production after being closed down for several years, while the Glückauf shaft operated by Kalichemie resumed production last year. A large modernisation programme in the Salzdetfurth mines provides for the underground crushing of the rock salts.

Government officials point out that, despite the loss of East German potash mines, which accounted for about 60% of Germany's entire prewar capacity, West German output last year equalled the entire German output in 1938. They say that the productivity of the mines has been considerably improved by the increases in the domestic and export sales and that this should be regarded as compensation for higher production costs.

## FRANCE

### More fertilisers

French output of phosphate fertilisers in 1951 totalled 2,552,500 tons, corresponding to a monthly average of 212,700 tons, against 193,100 in 1950 and 198,200 in 1938.

## ITALY

### Sulphur producers allege discrimination against sterling customers

Producers of refined sulphur have complained to the Ministry of Industry against the Government's policy on the licensing of sulphur exports. The Government has been withholding licences, they claim, for exports of refined sulphur to the sterling area in order to avoid further increases in the Italian sterling balances.

Sterling area countries have traditionally been Italy's best customers for sulphur, the producers say, and the Government's policy of switching exports is impracticable in view of the lower price of American sulphur. Refiners have also been particularly insistent that they should receive permits for exports to Greece and Egypt, from which countries demand is strong.

### New petroleum and chemical plant

The Butangas chemical group, one of the largest operating in the liquid fuel field in Italy, is to build a petroleum and chemical works on part of the site formerly occupied by the Caproni Aeroplane factory, now in liquidation. The plant will include a liquid fuel depot and some up-to-date installations for the manufacture of solvents and detergents. Equipment and machinery will be entirely of Italian design and manufacture.

## SWITZERLAND

### Nuclear Research Council set up

A Council of Representatives responsible for planning an international laboratory and organising other forms of co-operation in nuclear research has been set up with headquarters in Geneva, following a five-day conference organised by Unesco. Nine states have already signed the agreement setting up the Council: Denmark, France, Germany, Greece, Italy, the Netherlands, Sweden, Switzerland and Yugoslavia. Two others—Belgium and Norway—are expected to sign shortly. The Council will meet when at least five countries have ratified the agreement.

Each country will have one vote on the Council, but may send two delegates. This body will have an independent legal status and will be responsible for all future planning. It will also conclude a special agreement with Unesco and will co-operate with that organisation. Its budget will total \$200,000, to be contributed to by its member states. The Council's term of office will not exceed eighteen months, after which it will submit a report on the results of its work. It is expected that this will lead to a convention creating an international nuclear research laboratory, the location of which will be decided later.

The Council will also organise study groups to promote co-operation in nuclear physics and other groups to design the equipment for the laboratory.

The conference agreed that one task of the Council will be to organise a study



group at the Copenhagen Institute for Theoretical Physics. This group will survey the present situation in atomic physics with special regard to the work to be undertaken by European co-operation. It will also study problems which may be tackled with existing equipment and to be operated on a European basis. It will endeavour to stimulate European collaboration by furthering contacts between scientists in different countries and by offering possibilities for younger physicists to take part in the research work carried on in the Copenhagen Institute.

The Council is to consider an offer from Liverpool University to provide special facilities for a number of European physicists to work there. The university has agreed to take four experimental physicists for at least one year, two cyclotron engineers for a shorter period, and one or two theoretical physicists.

#### NORWAY

##### New galvanising plant

At the Tönsberg Harpoon Factory, South Norway, the country's biggest galvanising plant was opened recently. Instead of the usual iron or steel tank, the new plant is fitted with a firebrick tank, which the designer claims will reduce the waste of zinc very considerably. The zinc volume of about 100 tons is heated by a 320-kW electric element. The tank's galvanising capacity is about 40 tons a day or 12,000 tons a year, and the annual zinc consumption will be about 450 tons. The new galvanising plant will be able to handle large items which formerly had to be sent abroad for galvanising.

##### Iron ore project

The opening up of the Dunderland iron ore deposits in Nordland Province, North Norway, will take about five years and cost some £12,500,000, according to the director of the A/S Norsk Bergwerk, the Norwegian Government-owned mining company. The deposits are estimated to contain some 1,000,000,000 tons of iron ore and development work should begin when the construction of the nearby Mo i Rana iron and steel plant and the Rossaga hydro-electric station is completed.

#### PORTUGAL

##### New ammonium sulphate factories

Production of ammonium sulphate has started in two newly constructed factories in Portugal. Though this fertiliser was not previously produced in Portugal, the country's requirements are expected to be fully met when the factories come into full operation.

The larger of the factories is owned by the Companhia Uniao Fabril, Portugal's most important industrial concern. Output will be at least 40,500 tons annually when it comes into full operation and may rise to 45,000 tons if additional hydro-electric power becomes available. The cost

of the factory was 200,000,000 escudos. Equipment and technical assistance were provided by Britain.

The second factory has been established by a new company, the Amionaco Portugues; output is already running at a rate of 25,000 tons yearly now that the first stage of construction is completed.

#### DENMARK

##### Manganese sulphate from bog ore

Plans for the establishment of a plant in Jutland for the manufacture of manganese sulphate from bog ore are so well advanced that production is expected to start this autumn. The plant's estimated annual capacity is 1,000 tons of manganese sulphate, or double Denmark's total consumption of this product. About 500 tons of manganese sulphate will thus be available for export.

Denmark has recently exported considerable quantities of bog ore to the Ruhr industry. This ore has too low a manganese content to make extraction worthwhile, but the quality is considered good enough to supplement German stocks of high-quality iron ore.

In 1947, Denmark started to export bog ore for gas fortifying purposes. Since then exports have increased by 300%, amounting to 30,000 tons in 1951. The Danish authorities have now banned exports of this ore with a manganese content of more than 10% in order to conserve supplies of this quality for the domestic industry.

#### CANADA

##### Nickel production in top gear

Sales of nickel, nickel alloys, copper, platinum and other products by the International Nickel Co. of Canada Ltd. in 1951 are estimated at \$286,700,000, which compares with \$228,071,346 in 1950.

The company operated at maximum capacity in 1951. Emergency facilities were installed and put into operation several months ahead of schedule, bringing the rate of nickel production to 21,000,000 lb./month, an increase of 5% over the rate prevailing prior to mid-year.

At one and the same time, the company's extensive underground development programme initiated during World War 2 was pushed further toward completion. Total ore mined during the year from both underground and surface operations was 11,800,000 tons, compared with 9,849,000 tons in 1950. This increase of approximately 2,000,000 tons came entirely from underground ores. Production from underground in 1951 was 7,780,000 tons, the largest quantity hoisted in any of the 50 years of the company's history.

By the end of 1953, when production from surface operations will be fully replaced, the company will be able to hoist 13,000,000 tons of ore a year, which is twice as much from underground as in any year prior to 1951.

#### YUGOSLAVIA

##### Chemical developments

Work is to begin this year on the construction of a chemical combine at Sabac, Serbia. The first section to be built will be a sulphuric acid plant. This will use as its raw material the waste of gases extracted during the processing of copper ores from the Bor mines. Another sulphuric acid plant is to be integrated with an existing zinc electrolysis plant. Eventually, output from the two plants will go a long way towards covering the country's requirements. Meanwhile, superphosphate manufacturing facilities are being considerably extended.

A chemical plant at Zagreb will shortly begin manufacturing pectin in quantities sufficient to meet the country's requirements. Hitherto pectin has been imported, but supplies have been inadequate.

The Slovene firm, Silva-Product, which produced 5,000 kg. of essential oils in 1951, has begun producing pine pitch for the pharmaceutical industry. It is expected to make some 120 tons of this product during 1952.

#### PAKISTAN

##### Fertiliser factory planned

The Power-Gas Corporation Ltd., Stockton-on-Tees, confirm that they have been asked by the Government of Pakistan to submit proposals for the construction of a fertiliser factory in that country as a consequence of a comprehensive survey and report carried out by Power-Gas in conjunction with Chemical Construction Corporation, New York, some two years ago.

The proposed plant is intended to manufacture 50,000 tons of ammonium sulphate p.a. and the main raw materials to be employed are a non-caking coal and gypsum, both of which occur naturally and reasonably near a suitable factory site. It is proposed that the coal be gasified directly to produce a gas suitable, after purification, for ammonia synthesis and that ammonium sulphate be manufactured from this synthetic ammonia by the gypsum process.

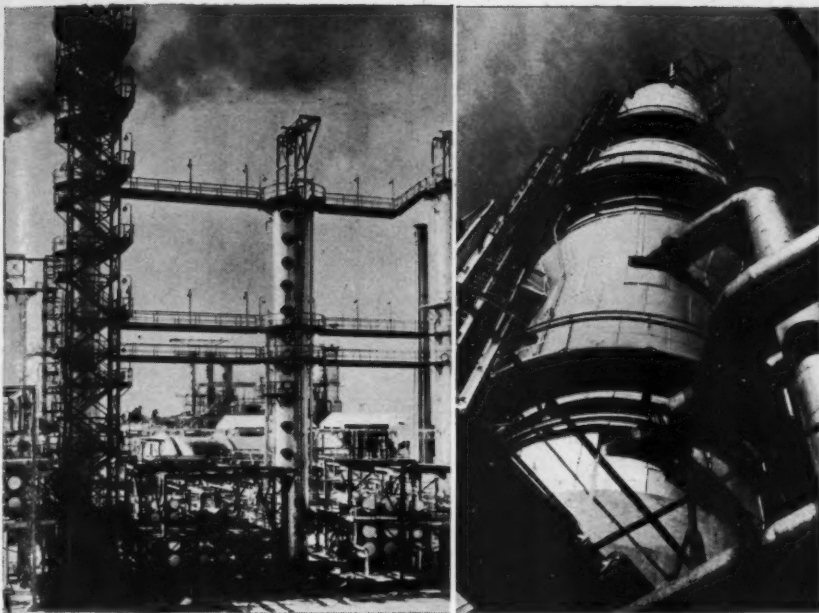
Power-Gas were the chief contractors for the Sindri fertiliser plant which was recently erected in India and which will be described in detail in our next issue.

#### INDIA

##### New dye and drug factory

A new dyestuffs and pharmaceutical plant was scheduled to start operations last month at Bulsar, near Bombay. The plant, which is claimed to be the first of its type in India, is being run by a new company, Atul Products Ltd., the formation of which was assisted by American Cyanamid Co., who subscribed 10% of the capital of the new company, the rest coming from Indians. American Cyanamid Co. are also providing technical assistance, technicians and some of the raw materials.

It is claimed that this plant will produce enough sulpha drugs to cure most of the



#### FRENCH REFINERY EXTENSIONS COST £8,000,000

Fractionating columns of the new reforming unit recently put into operation at the Berre oil refinery of the Cie de Raffinage Shell Berre near Marseilles (left). The other photograph shows the main fractionating column of the new crude distillation (topping) unit. These extensions, opened a few weeks ago, cost £8,000,000 and they make the Berre plant one of the largest refineries in France. The distillation unit has a capacity of 8,000 tons day and its outstanding features are six fractionating columns, one of which weighs 300 tons. The 1,200 tons/day reformer transforms part of the distillate into high-octane fuels, using a high-temperature cracking process. Berre refinery had a total intake in 1951 of 2,360,000 tons of crude oil against 1,140,000 tons in 1950; its annual capacity has now reached 3,000,000 tons, that is, treble the 1950 total and six times greater than immediately before the war. About 70% of the feedstock is obtained from Kuwait on the Persian Gulf, with smaller quantities from Iraq, Venezuela, France and Algeria. By the end of 1953 a modern fluid catalytic cracking unit will be in operation on the lines of those already operating at Stanlow (England) and Pernis (Holland). A Houdry catalytic cracking plant is already used at Berre for aviation and motor spirit.

infectious diseases which seriously hamper India's economic progress. Sulphadiazine, which has proved its effectiveness against plague, will be produced first. The vitamin folic acid and the antibiotic aureomycin will be produced shortly afterwards. The plant will, it is stated, also produce enough of the 20 to 25 more common dye-stuffs to supply the country's textile, paper and other industries. At the outset, the plant will save India \$500,000 p.a. in foreign exchange. These savings are expected to increase considerably as the plant reaches full production.

#### KUWAIT

##### Largest water distillation plant

Designed to produce 1,000,000 gal. of fresh water a day from sea water, a new distillation plant is to be built outside the town of Kuwait at the expense of the State. The design will provide for future expansion to five times this size. The world's largest water distillation plant is the Kuwait Oil Co.'s plant at its loading port, Menael-Ahmadi. This plant is smaller than the one planned, distilling 600,000 gal./day from the sea, of which 200,000 gal. are piped daily to the town of Kuwait. The remainder serves the oil company's estab-

lishments, which, in effect, are a self-contained town separated from Kuwait town by more than 20 miles of clean hard sand. This plant uses spent steam from the oil company's power generators. The 1,000,000-gal. plant will be fired by oilfield gas.

Water is Kuwait's first need. The territory, some 6,000 square miles, is a sandy desert in which even the artesian well water is salt. No river touches the state. The town's traditional supply is brought from the Shatt-el-Arab in dhows in which the water-sellers load their skins. The new distillation plant and many other improvements in the country's amenities are being made possible by the development of the Kuwait oilfield, one of the largest in the world, from which it is expected to produce 40,000,000 tons this year, on which royalties and taxes of £50,000,000 are payable.

#### MEXICO

##### Big refinery developments

Work has started on the building of a 173,000,000-peso lubricants plant, at the Salamanca refinery, operated by Petroleos Mexicanos (Pemex). The plant will go into production early in 1954, and will produce 2,200 metric tons of lubricant

daily. Foreign lubricants will be required in very small quantities, if at all, after the completion of this plant.

Another Pemex refinery, under construction at Ciudad Madero, will be completed by the end of the year. Half of the 40,000,000 pesos to be spent will be employed for thermo-electric generating plant, construction of which started at the end of January. More recently building operations started on a cracking plant with a capacity of 20,000 barrels daily, and on a secondary distillation plant.

Refineries will also be built at the ports of Guaymas, Mazatlan and Salina Cruz. In addition, sulphur extraction plants are being built at the Azcapotzalco refinery and at Ciudad Madero.

#### U.S.A.

##### Sulphur production up

The Texas Gulf Sulphur Co. has announced in its annual report a change in export procedure. Pursuant to directives of the National Production Authority, it is stated, the company supplies approximately 66% of the sulphur exported through the Sulphur Export Corp'n. However, agreement was reached with the Freeport Sulphur Co. to terminate the activities of the Sulphur Export Corp'n. with shipments to fulfil allocations for the first quarter of 1952. All export shipments by Texas Gulf are now handled directly by the company.

The report also states that at the company's principal producing property, production continues at approximately 150% of normally expected capacity. Near Beaumont, Texas, construction of an operating plant is nearing completion and it is hoped that this plant will be in production during the second quarter of 1952. Exploration in the Republic of Mexico which began in 1950 is still in progress, but to date no discovery has been made.

##### Catalyst production increased

The American Cyanamid Co. has begun a major expansion of production facilities in its recently constructed micro-spheroidal cracking catalyst plant near Michigan City, Indiana. The expansion will be completed by midsummer, raising the plant's production rate to a level sufficient for a daily 800,000 barrels of refining capacity. The company, which pioneered the development in 1945 and commercial production in 1946 of the micro-spheroidal catalyst, has been the world's major supplier of the product, producing more than four times as much of the catalyst as any other firm.

Expansion of the Michigan city plant, which was constructed last August, is the result of a record high demand for high-octane fuels. The company also operates similar facilities in Fort Worth, Texas.

##### Fertiliser supply prospects

The U.S. Agriculture Department predicts that more nitrogen and potash but less phosphate will be available for ferti-

lisers in 1952. The Department told a meeting of the Fertiliser Industry Advisory Committee that supplies of basic fertilisers for 1952 would probably be: Nitrogen, 1,375,000 tons, up 7% over 1951; potash, 1,515,000 tons, up 5%; and phosphates, 2,100,000, 6% lower than 1951.

Members of the industry group said the shortage of sulphuric acid for phosphates production was their leading problem. They questioned whether everything was being done to increase sulphur supplies and urged steps to be taken to secure the critically short commodity from Mexico, Central America and other areas.

#### Record paint sales

The U.S. paint industry sold a record value of \$1,181,000,000 worth of its product in 1951, according to the National Paint, Varnish and Lacquer Association. The total is 4.7% above the 1950 value. These figures were based on reports from firms representing 87% of the industry. On an adjusted basis, 1951 sales stood at \$1,357,000,000.

#### Record plastics output

Sales of plastic materials in the U.S. during 1952 are expected to exceed the record 2,146 million lb. moved during 1951, according to the vice-president of the Bakelite Co. He pointed out that the 1951 figure was about three times the amount sold in 1946, the first post-war year. The vinyls, mostly polyvinyl chloride and its copolymers, had shown immense gains in usage. Over 400 million lb. were sold last year compared with an output of only 4,000,000 lb. in 1936. Sales of phenolics were also close to 400 million lb., he said.

There had been a sharp increase in sales of styrene resins, urea and melamine-based products, but a small decline had occurred in the use of cellulose-based plastics due to competition from the cheaper polystyrenes. This did not mean that cellulose were 'wiped out,' he said, and success will be attained in finding applications where their toughness and impact resistance would justify the higher price.

## Meetings

#### Institution of Chemical Engineers

May 3. 'Stainless Steel in Chemical Plant,' by A. M. Hucheson, 3 p.m., Latin Theatre, The University, Edmund Street, Birmingham.

#### GRADUATES' AND STUDENTS' SECTION

April 17. Centre general meeting followed by technical film show, 7 p.m., Shell Refining & Marketing Co. Ltd., Thornton Research Centre.

April 18. 'The Fluid Catalytic Cracking Process,' by J. G. Lucas, 6.30 p.m., Caxton Hall, Westminster, London, S.W.1.

April 29. Centre annual meeting followed by 'The Application of Planned Maintenance in Chemical Industries,' by R. K. B. Lyne, 7 p.m., Room 121, University College of South Wales and Monmouthshire, Museum Avenue, Cathays Park, Cardiff.

#### Society of Chemical Industry

April 22-24. Conference on 'Adhesives,' Beveridge Hall, University of London, W.C.1.

May 5. 'Laboratory Fractionation,' by Dr. E. F. G. Herington, 6.30 p.m., Chemical Society, Burlington House, Piccadilly, London, W.1.

May 6. Chemical Engineering Group. 'Chemical Engineering and Atomic Energy,' by C. M. Nicholls and A. S. White, 5.30 p.m., Burlington House, Piccadilly, London, W.1.

#### Chemical Society

April 11. 'Mechanism of Oxidation of Organic Compounds,' by Dr. W. A. Waters, 5.15 p.m., Chemistry Department, United College, St. Andrews.

April 18. 'Polyacetylenes,' by Prof. E. R. H. Jones, 5.15 p.m., Chemistry Department, United College, St. Andrews. Joint meeting with R.I.C.

April 23. 'The Chemical Exploration

of the Stratosphere,' by Prof. F. A. Paneth, 6.30 p.m., the University, Manchester.

April 25. 'Acetylenic Compounds,' by Prof. E. R. H. Jones, 5.30 p.m., University College of Swansea.

May 2. 'Recent Advances in the Chemistry of the Heaviest Elements,' by Dr. J. S. Anderson, 5.30 p.m., King's College, Newcastle-upon-Tyne.

#### Royal Institute of Chemistry

April 7-8. Symposium, 'Electrolytic Processes in Chemical and Metallurgical Industries,' 5 p.m. and 4 p.m., respectively, University College, Gower Street, London, W.C.1. Joint meeting with S.C.I.

April 10. Annual general meeting followed by 'Some Problems of Antibiotic Production,' by A. L. Bacharach, 7 p.m., Municipal Technical College, Kingston-upon-Hull.

April 17. 'Some Aspects of the History of Science,' by Dr. F. Sherwood Taylor, 8 p.m., Luton Town Hall. Joint meeting with Luton Scientific Association.

#### Institute of Fuel

April 16. Annual general meeting followed by 'Thermal Factors in Furnace Design,' by Prof. R. J. Sarjant and W. Hulse, 2 p.m. (luncheon 1 p.m.), Engineers' Club, Manchester.

April 22. 'Recent Developments in Instruments,' by E. C. Klepp, 5.30 p.m., Institution of Mechanical Engineers, Storey's Gate, London, S.W.1.

May 5. Annual general meeting followed by a film show, 7.30 p.m., the University, Edmund Street, Birmingham.

#### Institute of Metals

April 18. 'Corrosion of Iron and its Prevention by Deposits of Non-Ferrous Metals,' by U. R. Evans, 6.30 p.m., the University, St. George's Square, Sheffield. Joint meeting with Institute of Metal Finishing.

#### Institute of Metal Finishing

April 23-26. The 1952 spring conference, the Grand Hotel, Eastbourne.

#### Institute of Petroleum

April 9. 'Corrosion Problems in Engineering,' by S. F. Dorey, 5.30 p.m., Manson House, 26 Portland Place, London, W.1.

#### Institution of Works Managers

April 22. 'Atoms,' by P. B. Moon, 7 p.m., Grand Hotel, Birmingham.

April 25. 'Incentives,' by T. Adams (Courtaulds Ltd.) and J. Lang (Triplex Safety Glass Co. Ltd.), 7.30 p.m., Imperial Hotel, Birmingham. Joint meeting with Incorporated Plant Engineers.

#### Oil and Colour Chemists Association

April 7. 'Materials Handling,' by C. C. Mell, 7 p.m., Royal Station Hotel, Kingston-upon-Hull.

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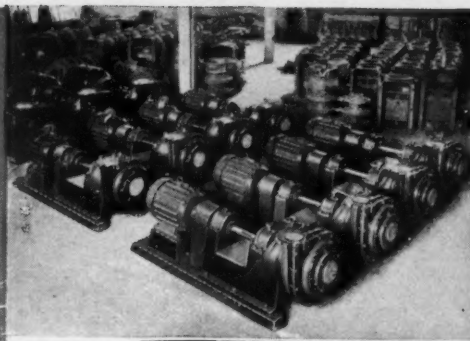
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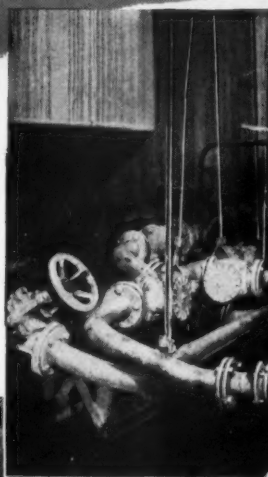
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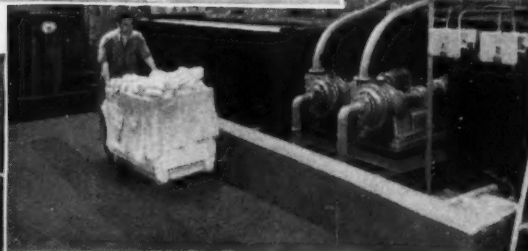
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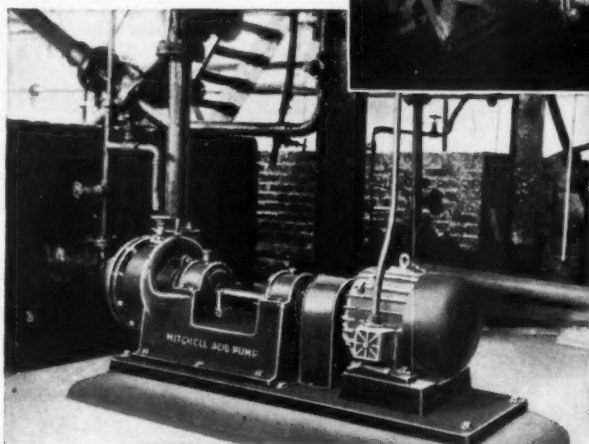
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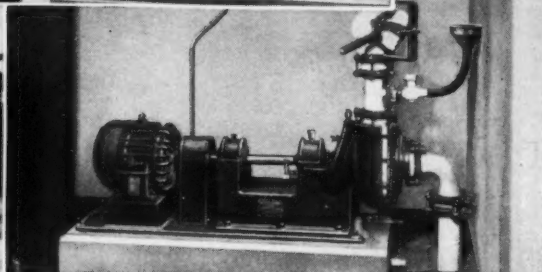
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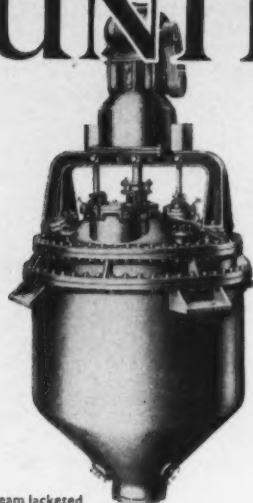
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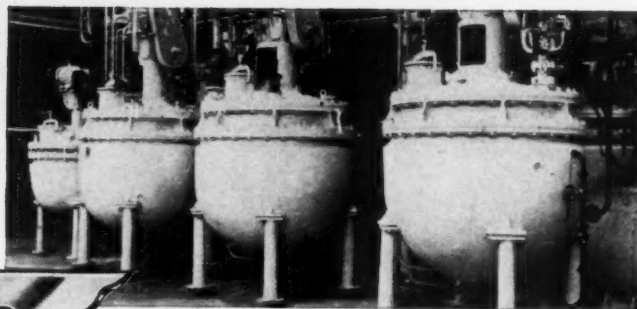
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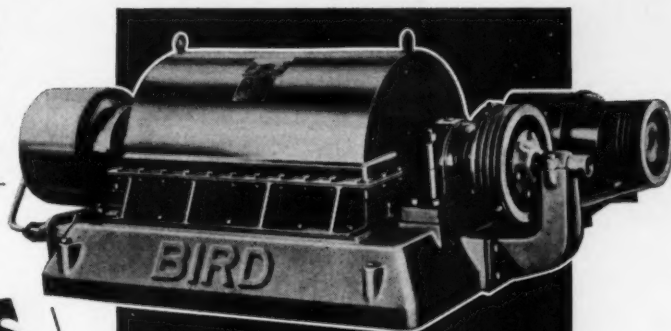
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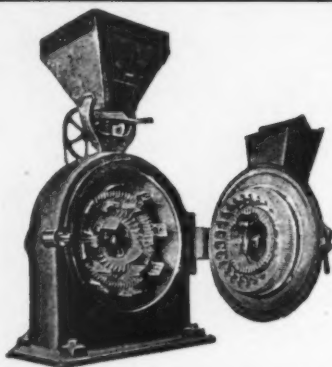
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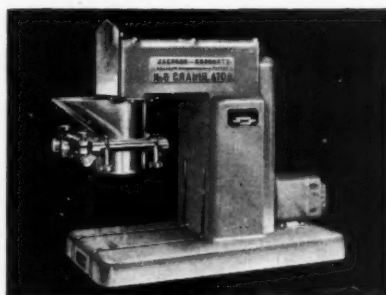
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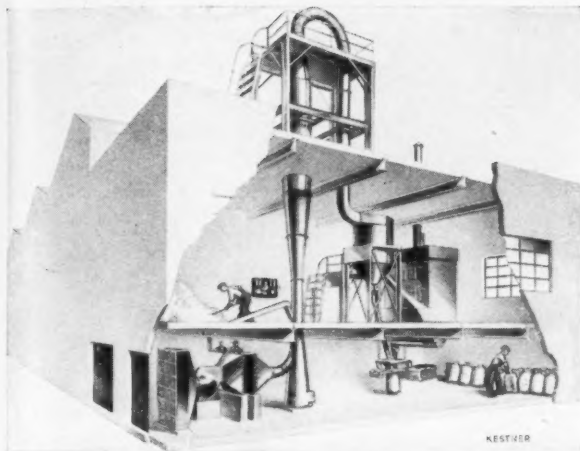


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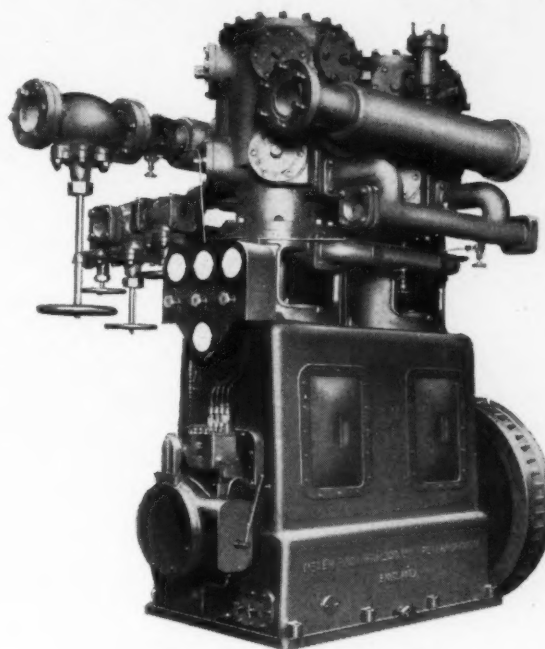
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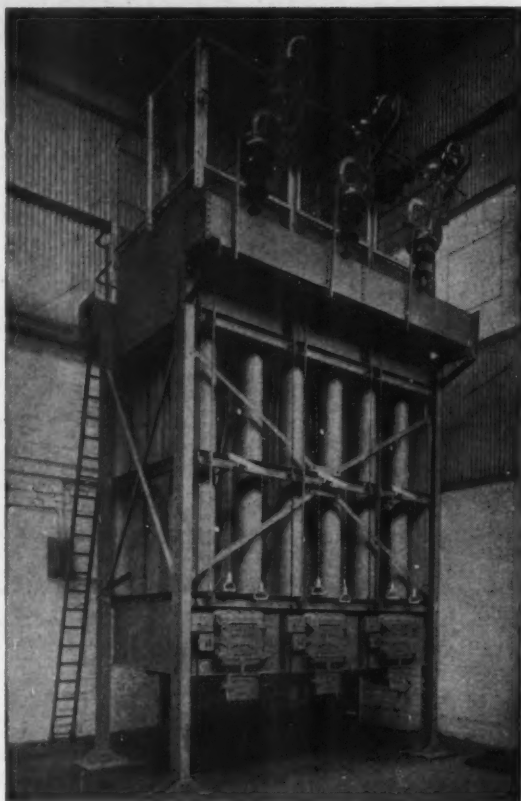
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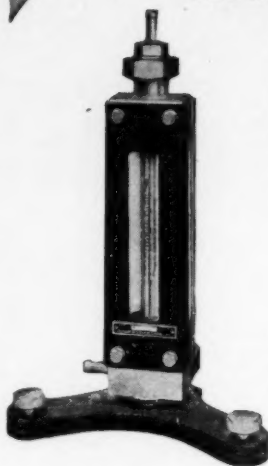
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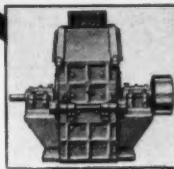


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